

# Inorganic Chemistry

including bioinorganic chemistry

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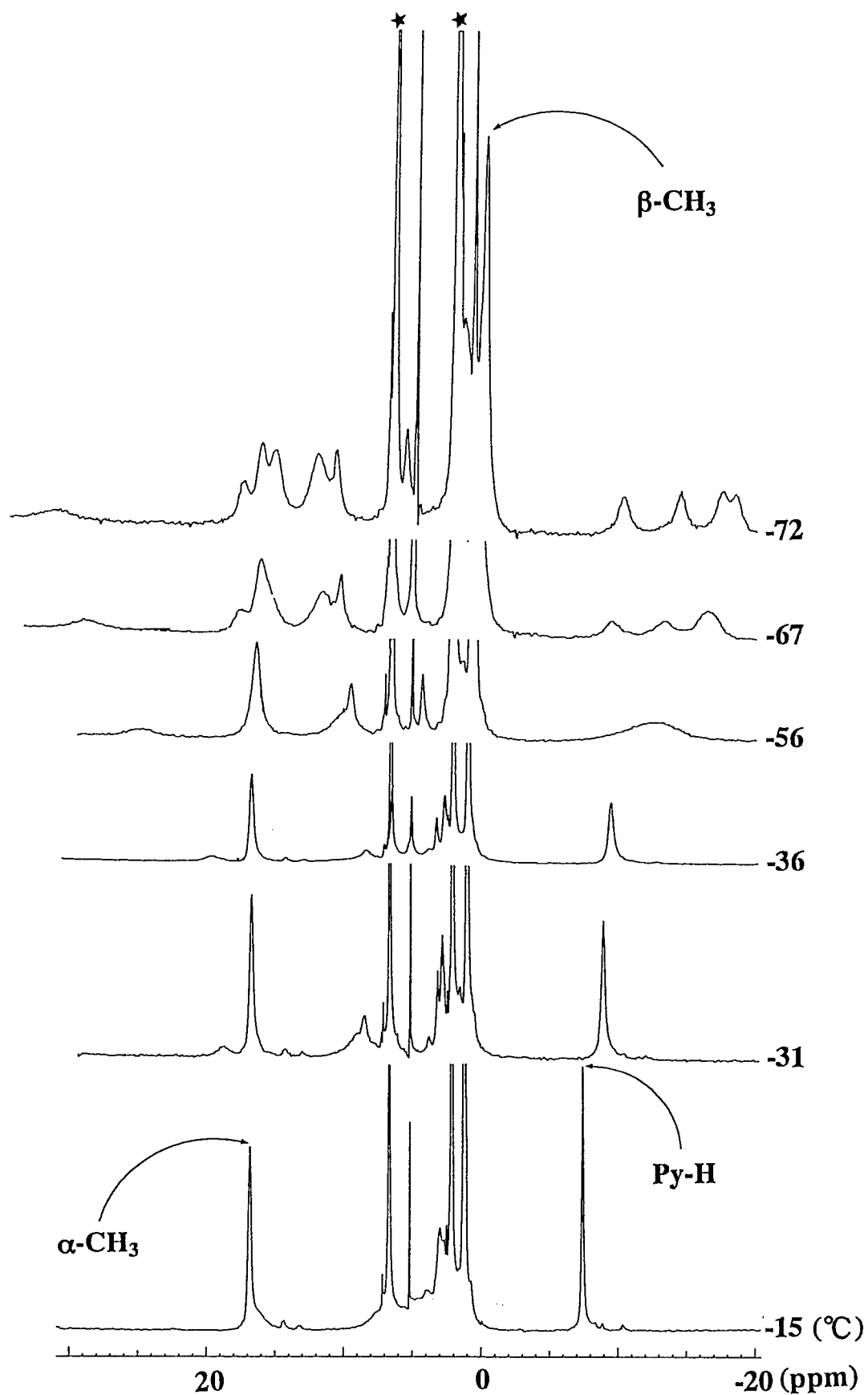
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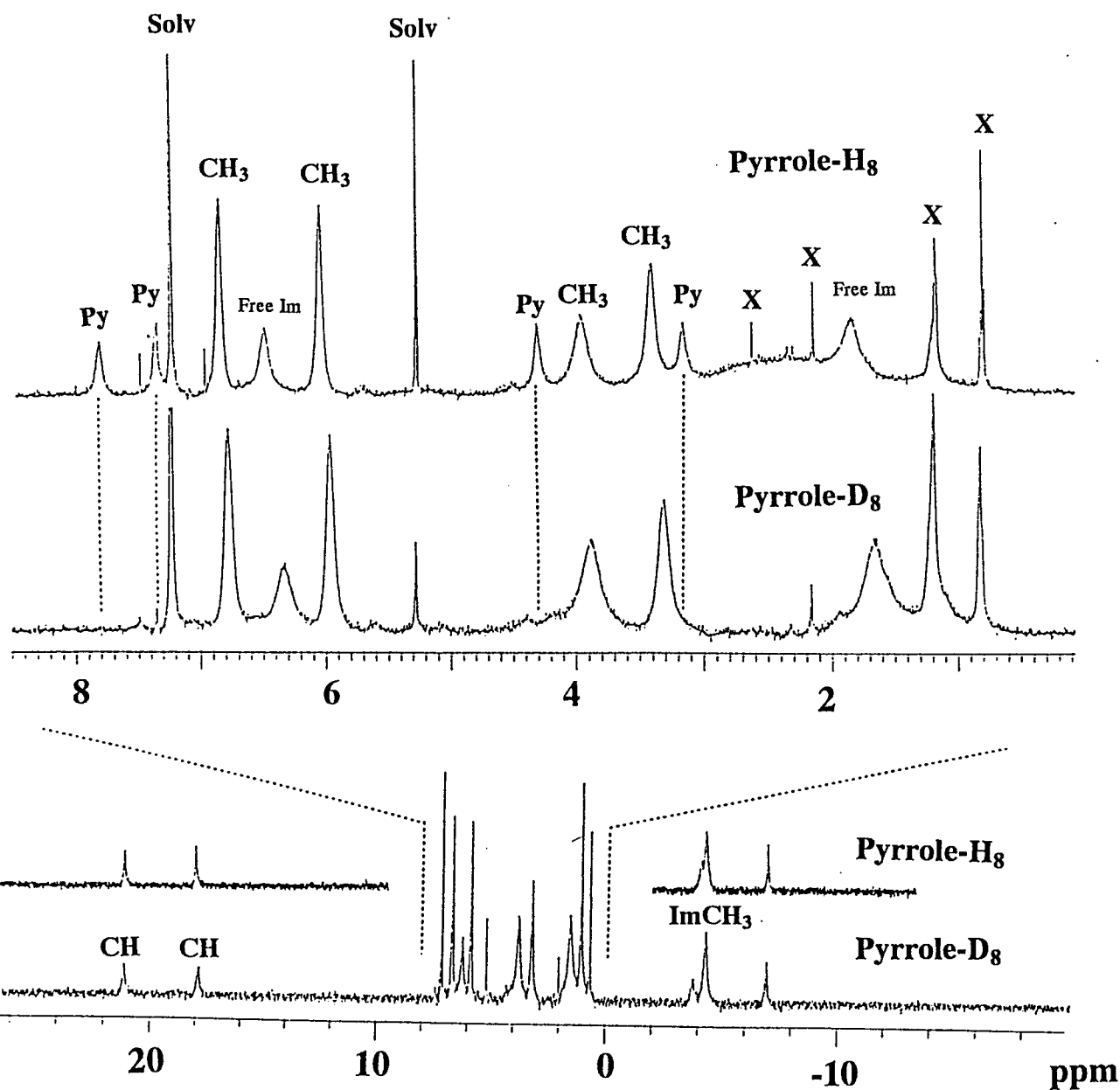
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## Supporting Information (1)

Temperature dependent  $^1\text{H}$  NMR spectra of  $[\text{Fe}(\text{TEtP})(2\text{-MeIm})_2]\text{Cl}$  in  $\text{CDCl}_3/\text{CD}_2\text{Cl}_2$  solution taken by JEOL FX90Q operating at 89.55 MHz. Signals for free 2-MeIm are signified by the asterisk(\*).



## Supporting Information (2)

$^1\text{H}$  NMR spectra of  $[\text{Fe}(\text{TiPrP})(2\text{-MeIm})_2]\text{Cl}(\text{py-H}_8)$  and  $[\text{Fe}(\text{TiPrP})(2\text{-MeIm})_2]\text{Cl}(\text{py-D}_8)$  in  $\text{CDCl}_3/\text{CD}_2\text{Cl}_2$  solution taken at  $-35^\circ\text{C}$  by JEOL GX400 operating at 399.65 MHz.

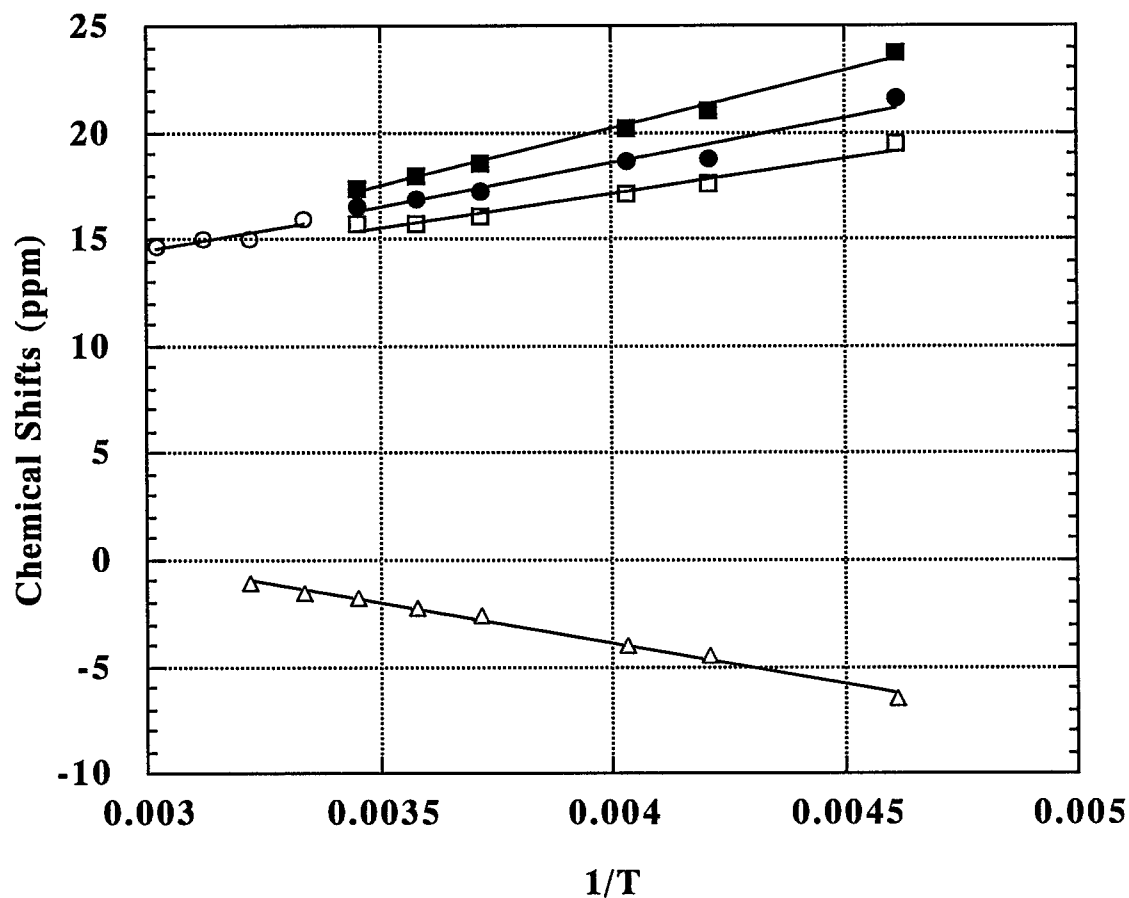
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## Supporting Information (3)

Temperature dependence of some proton signals of  $[\text{Fe}(\text{TiPrP})(2\text{-MeIm})_2]\text{Cl}$  taken in  $\text{CDCl}_3$ .

- one of the isopropyl methine signals at low temperature where methine protons gave two signals.
- another isopropyl methine signal at low temperature where methine protons gave two signals.
- average of the two isopropyl methine signals at low temperature.
- isopropyl methine signal at high temperature where two signals coalesced to give single line.
- △ methyl signal of the coordinated 2-MeIm.



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## Supporting Information (4)

## Synthesis

**meso-Tetraisopropylporphyrin, (TiPrP)H<sub>2</sub>:** A propionic acid (330 ml) solution containing pyrrole (4.07 g, 61 mmol) and isobutylaldehyde (3.18 g, 44 mmol) was heated at 110 °C for 2 h with stirring. The reaction mixture was cooled and most of the propionic acid was distilled off under reduced pressure. Chloroform (100 ml) was then added to the reaction mixture and the chloroform solution was washed twice with 20 ml of water and then with 20 ml of aq. NaOH. The organic layer, after dried on anhydrous Na<sub>2</sub>SO<sub>4</sub>, was treated with p-chloranil (1.0 g) at 61 °C for 1 h. The solvent was evaporated and the resulting solid was chromatographed on silica gel. Elution with CH<sub>2</sub>Cl<sub>2</sub> gave 340 mg of pure material. <sup>1</sup>H NMR(CDCl<sub>3</sub>, δ ppm, 25°C): 2.27(CH<sub>3</sub>), 5.25 (CH), -1.8 (NH), 9.39 (py-H).

**meso-Tetraethylporphyrin, (TEtP)H<sub>2</sub>,** was similarly prepared by heating pyrrole (4.07 g, 61 mmol) and propionaldehyde (2.40 g, 41 mmol) in propionic acid (330 ml) at 100 °C. Chromatography on silica gel yielded 240 mg of pure material. <sup>1</sup>H NMR (CDCl<sub>3</sub>, δ ppm, 25°C): 2.01 (CH<sub>3</sub>), 4.94(CH<sub>2</sub>), -2.3 (NH), 9.43 (py-H).

**meso-Tetramethylporphyrin, (TMeP)H<sub>2</sub>,** was similarly prepared by heating pyrrole (4.07 g, 61 mmol) and acetaldehyde (0.87 g, 20 mmol) in propionic acid (330 ml) at 90 °C. Chromatography on silica gel yielded 125 mg of pure material. <sup>1</sup>H NMR(CDCl<sub>3</sub>, δ ppm, 25°C): 4.53(CH<sub>3</sub>), -2.5 (NH), 9.48 (py-H).

**meso-Tetraisopropylporphyrinatoiron(III) chloride, [Fe(TiPrP)]Cl:** An acetic acid (30 ml) solution containing tetraisopropylporphyrin (50 mg, 104 μmol), FeCl<sub>3</sub>•6H<sub>2</sub>O (50 mg, 185 μmol), and sodium acetate (7.5 mg, 91 μmol) was refluxed for 2 h. To the cooled reaction mixture was added 100 ml of CHCl<sub>3</sub>. The CHCl<sub>3</sub> solution was washed twice with 20 ml of water and then with 20 ml of dil. HCl. The organic layer was dried on anhydrous Na<sub>2</sub>SO<sub>4</sub>. The solvent was evaporated and the resulting solid was chromatographed on silica gel. Elution with CH<sub>2</sub>Cl<sub>2</sub>-CH<sub>3</sub>OH(5%) gave purple solid which was further purified by recrystallization from CH<sub>2</sub>Cl<sub>2</sub> and hexane. <sup>1</sup>H NMR(CDCl<sub>3</sub>, δ ppm, 25°C): 9.5(CH<sub>3</sub>), 28.9(CH), 90.5(py-H).

**meso-Tetraethylporphyrinatoiron(III) chloride, [Fe(TEtP)]Cl,** was similarly prepared. <sup>1</sup>H NMR(CDCl<sub>3</sub>, δ ppm, 25°C): 7.2(CH<sub>3</sub>), 62.1(CH<sub>2</sub>), 88.6(py-H).

**meso-Tetramethylporphyrinatoiron(III) chloride, [Fe(TMeP)]Cl,** was similarly prepared. <sup>1</sup>H NMR(CDCl<sub>3</sub>, δ ppm, 25°C): 127.6(CH<sub>3</sub>), 87.6(py-H).