

Electronic Supplementary Information

Stepwise synthesis of heterotrimetallic Fe^{II}/Pd^{II}/Au^I coordination cages

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1 General

All reagents were obtained from commercial sources and used without further purification unless stated otherwise. Complex **1** and complex **2** were synthesized in analogy to reported procedures.^{1,2}

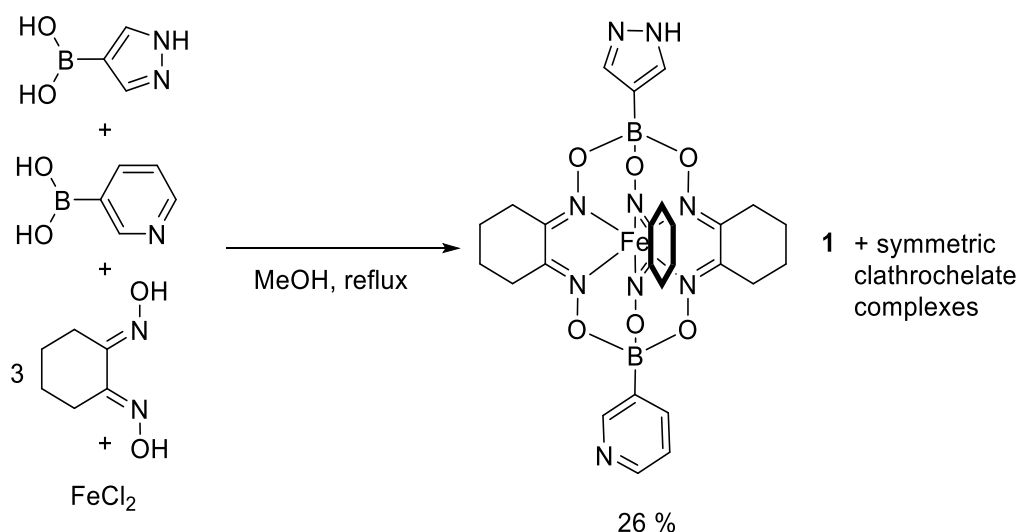
NMR spectra were measured on a Bruker Avance III HD spectrometer (¹H: 600 MHz, ¹³C: 151 MHz) equipped with a CPPBBO_z 5 mm probe a Bruker Avance III spectrometer (¹H: 400 MHz, ¹³C: 101 MHz) equipped with a BBFO_z 5 mm probe and a Bruker Avance III spectrometer (¹H: 400 MHz, ¹³C: 101 MHz) equipped with a Prodigy BBO 5 mm cryoprobe. The chemical shifts are reported in part per million (ppm) using the solvent residual signal as a reference. The DOSY analysis was performed using the the Peak Height Fit DOSY transform from *MestrelNova*. The solvodynamic radii were calculated from the average diffusion coefficient, as determined by *MestrelNova*. The values were normalized using the solvent as an internal standard.³ Literature values were used for the viscosity and the diffusion coefficient of DMSO at 298 K.^{4,5}

Mass spectrometry analyses were performed on a LTQ Orbitrap FTMS instrument (LTQ Orbitrap Elite FTMS, Thermo Scientific, Bremen, Germany) operated in the positive mode coupled to HESI-II probe in an Ion Max ion Source. Samples were injected at an infusion rate of 10 µL/min. The experimental conditions for the ionization voltage was 1.2 kV whereas the temperature of ion transfer capillary was 80 °C. FTMS spectra were obtained using the high mass range between 200–4000 *m/z* range in the reduced profile mode with a resolution set to 120 K. In all spectra, one microscan was acquired with a maximum injection time value of 1000ms. Samples were diluted in ACN to a total concentration of around 0.05 mM.

The models of cage **4** and **5** were constructed using *Spartan* and it's MMF energy minimization.

2 Syntheses

2.1 Synthesis of complex 1



Scheme S1. Synthesis of complex 1.

Nioxime (200 mg, 1.4 mmol), pyridin-3-ylboronic acid (65 mg, 0.51 mmol), 4H-pyrazoleboronic acid (59 mg, 0.49 mmol) and anhydrous FeCl_2 (60 mg, 0.49 mmol) were dissolved in MeOH (35 mL) and heated under reflux for 4 h under an atmosphere of N_2 . The solvent was removed under reduced pressure. The product was dissolved in a minimum amount of CHCl_3 and purified by column chromatography with a mixture of $\text{CHCl}_3/\text{MeOH}/\text{triethylamine}$ (99:1:0.05) as eluent. Yield 88 mg, 26%. ^1H NMR (400 MHz, CD_2Cl_2) δ 8.79 (s, 1H), 8.51 (dd, $J = 4.9, 1.9$ Hz, 1H), 7.92 (dt, $J = 7.5, 1.9$ Hz, 1H), 7.65 (s, 2H), 7.23 (ddd, $J = 7.5, 4.9, 1.0$ Hz, 1H), 3.07 – 2.74 (m, 12H), 1.81 (m, $J = 3.0$ Hz, 12H). ^{13}C NMR (101 MHz, CD_2Cl_2) δ 153.29, 152.21, 149.25, 139.62, 123.20, 26.60, 21.99. (C-B pyrazole and C-B pyridine not detected). ESI-MS: m/z calculated for $\text{C}_{26}\text{H}_{32}\text{B}_2\text{FeN}_9\text{O}_6^+$ $[\text{M}+\text{H}]^+$ 644.2006, found 644.2024.

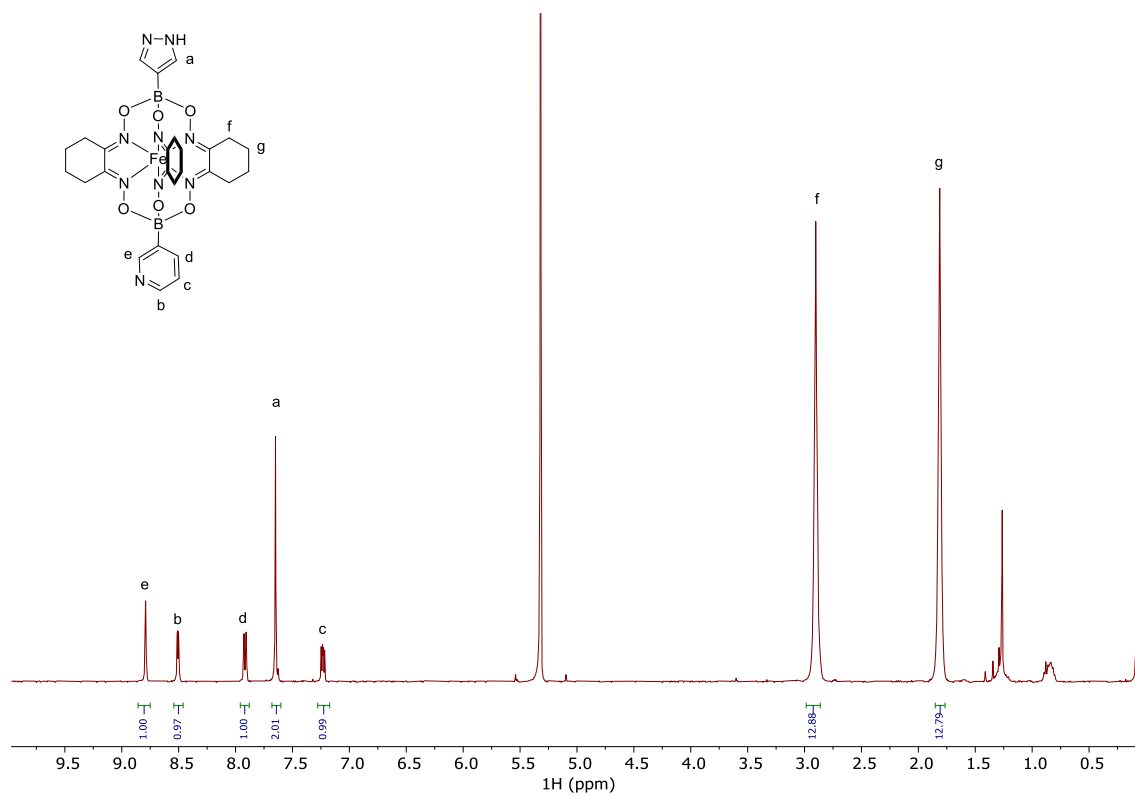


Figure S1. ^1H NMR spectrum (400 MHz, CD_2Cl_2 , 298 K) of complex 1.

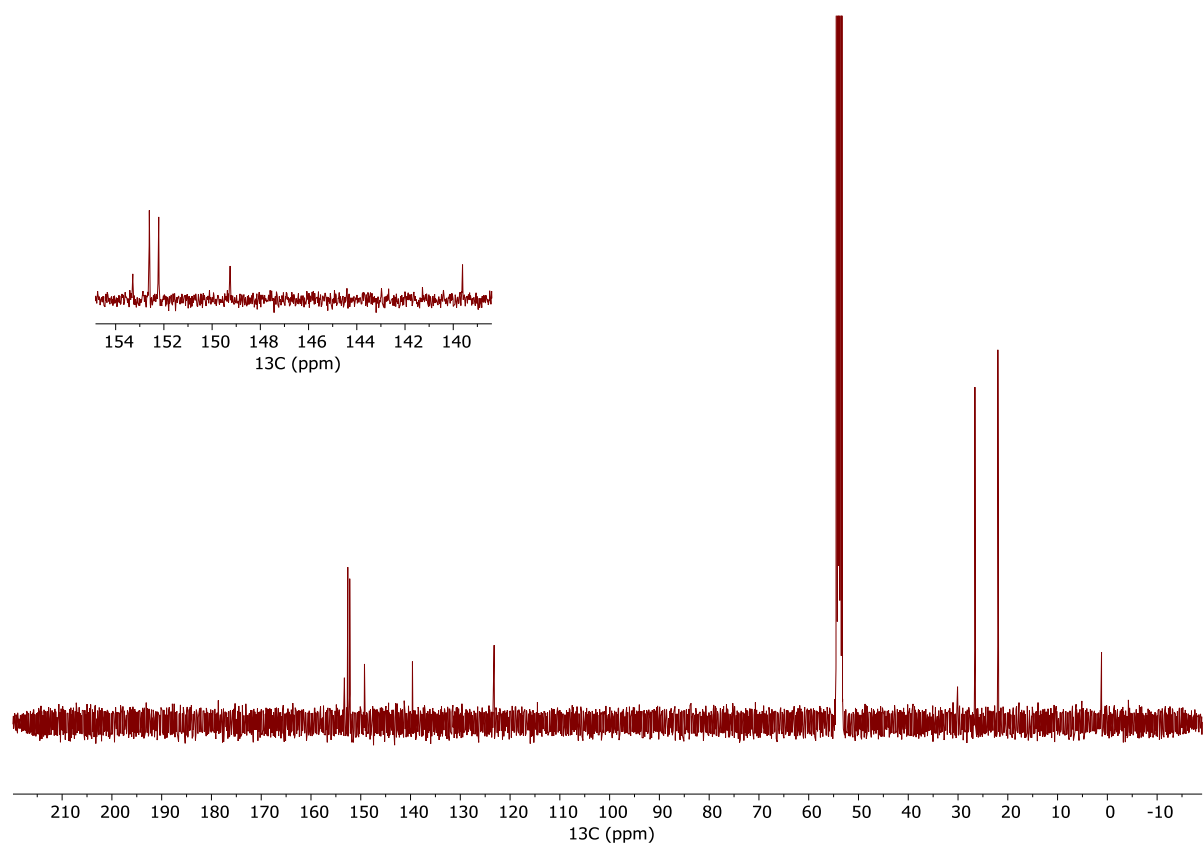


Figure S2. ^{13}C NMR spectrum (101 MHz, CD_2Cl_2 , 298 K) of complex 1.

2.2 Synthesis of complex 2

Triethylamine (50 μ L, 0.16 mmol) was added to a stirred solution of AuCl(SMe₂) (17.3 mg, 0.06 mmol) and complex 1 (38 mg, 0.059 mmol) in THF (5 mL). The mixture was stirred for 18 h at RT under an atmosphere of N₂. Subsequently, MeOH (approximately 25 mL) was added to precipitate the product and to dissolve the ammonium salt. The product was isolated by centrifugation and dried under vacuum. Yield: 43 mg, 87%. ¹H NMR (400 MHz, CD₂Cl₂) δ 8.80 (s, 1H), 8.51 (dd, *J* = 4.9, 1.9 Hz, 1H), 7.96 – 7.88 (m, 1H), 7.68 (s, 2H), 7.27 – 7.19 (m, 1H), 2.96 – 2.89 (m, 12H), 1.82 (t, *J* = 3.9 Hz, 12H). ¹³C NMR (151 MHz, CD₂Cl₂) δ 152.55, 151.78, 151.46, 148.52, 142.33, 138.70, 25.79, 21.16 (C-B pyrazole and C-B pyridine not detected). ESI-MS: *m/z* calculated for C₇₈H₉₃Au₃B₆Fe₃N₂₇O₁₈³⁺ [M+3H]³⁺ 840.1593, found 840.1604.

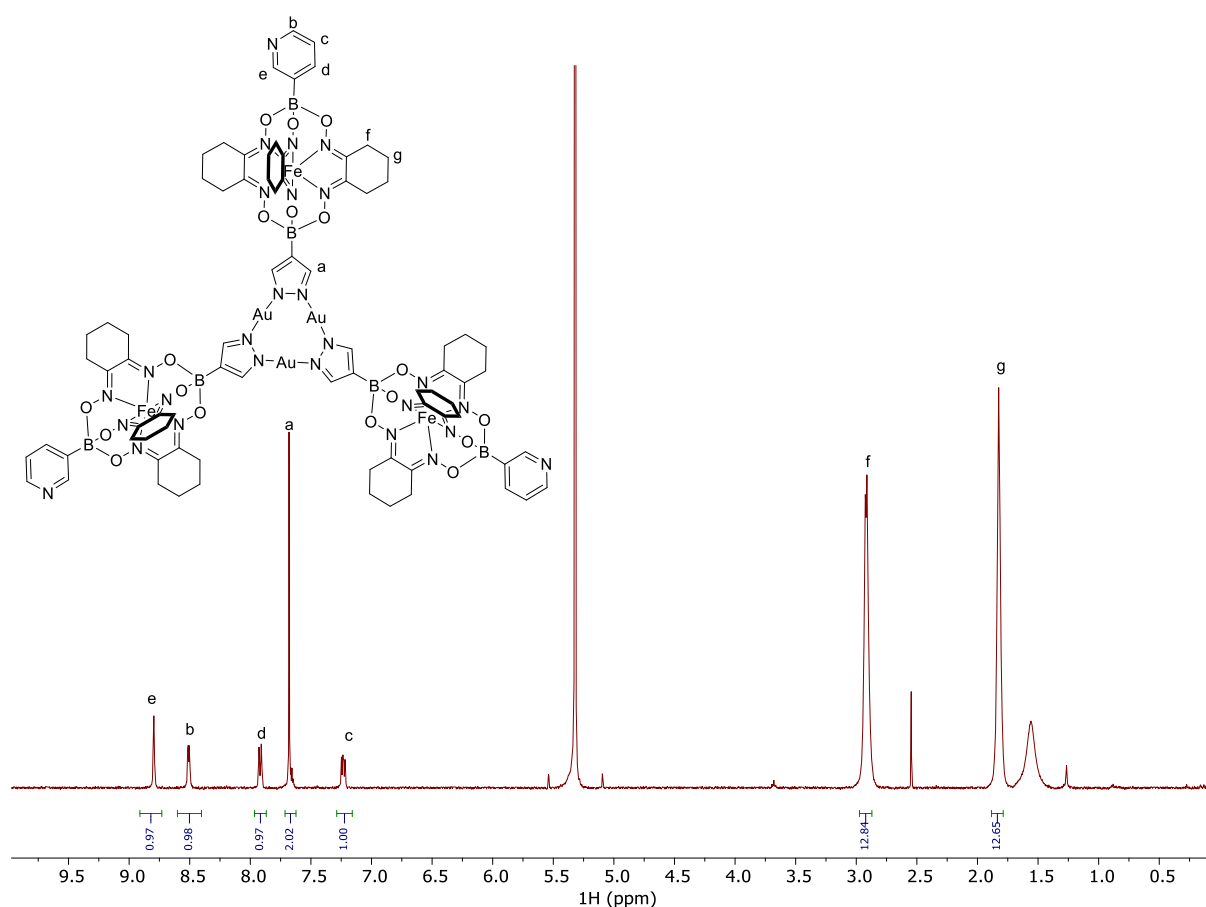


Figure S3. ¹H NMR spectrum (400 MHz, CD₂Cl₂, 298 K) of complex 2.

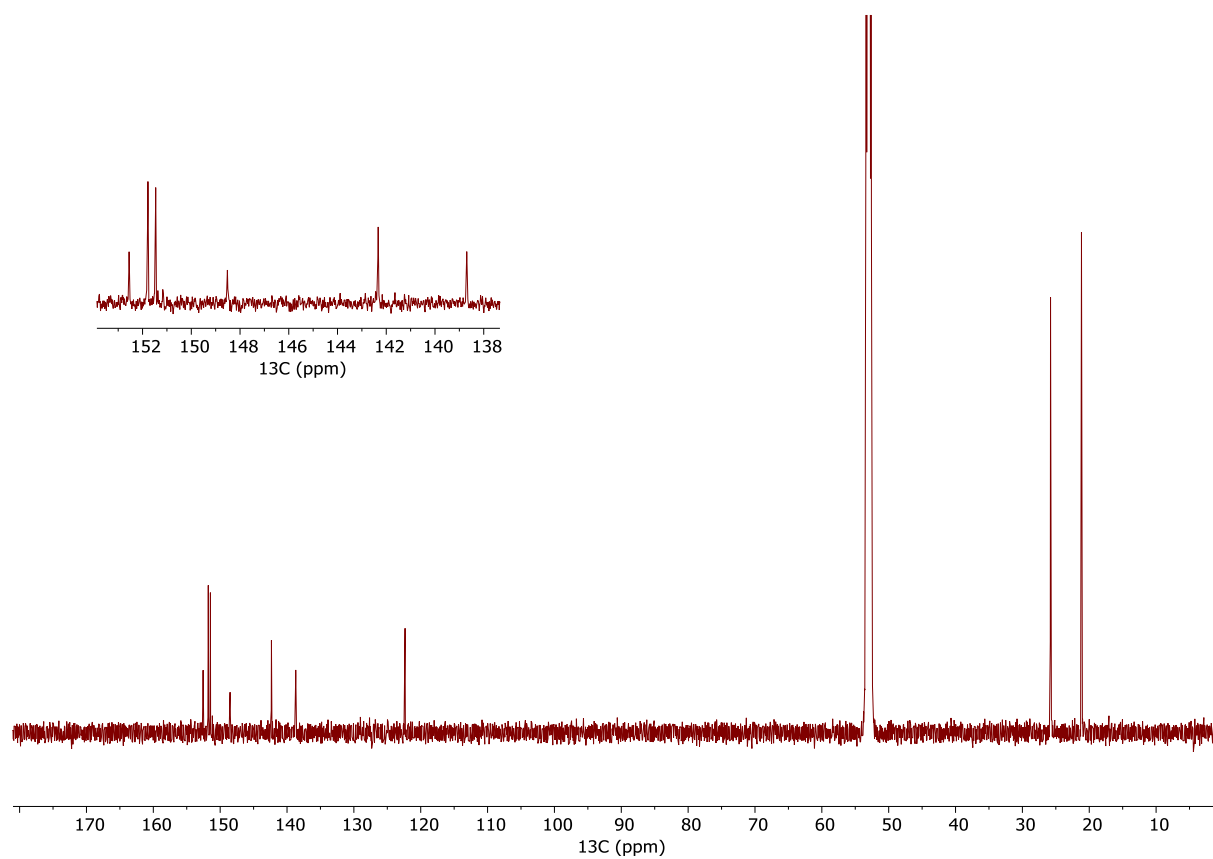


Figure S4. ^{13}C NMR spectrum (151 MHz, CD_2Cl_2 , 298 K) of complex **2**.

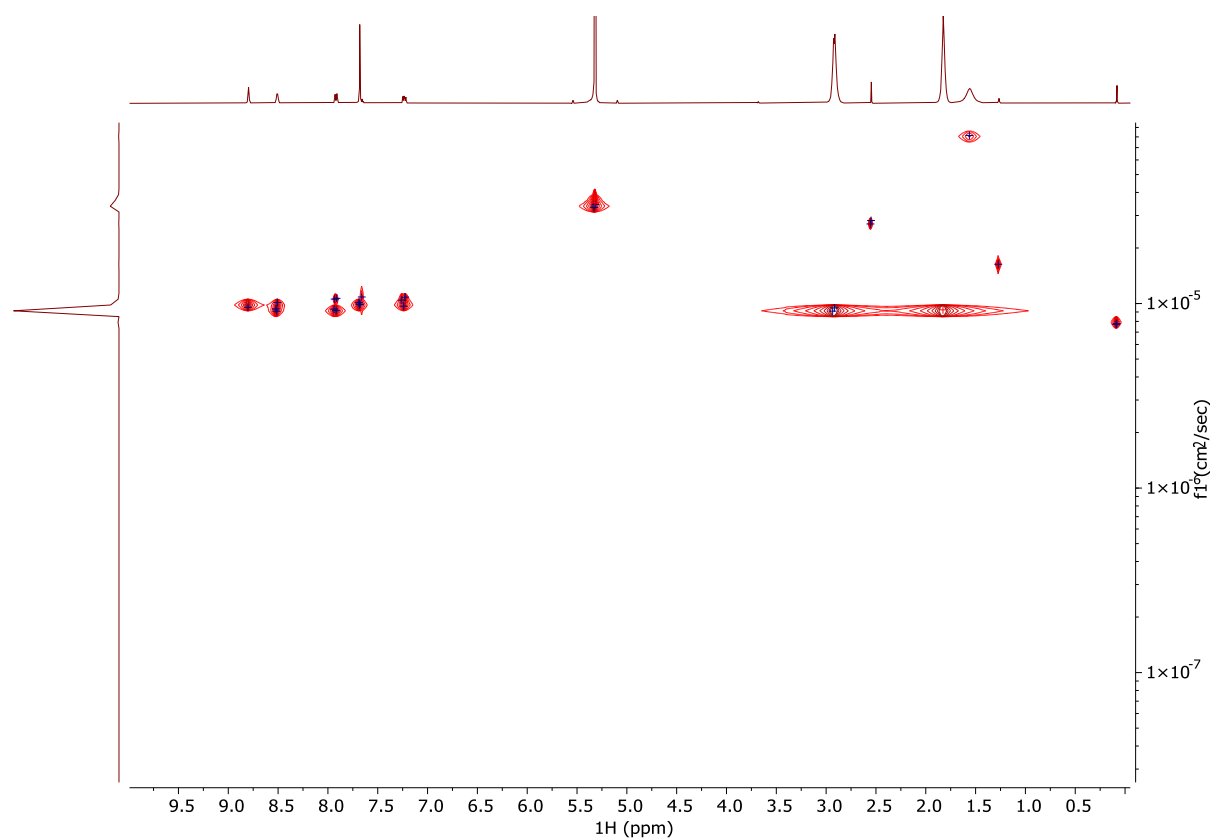


Figure S5. ^1H - ^1H DOSY NMR spectrum (400 MHz, CD_2Cl_2) of complex **2** ($D = 9.14 \times 10^{-10} \text{ m}^2\text{s}^{-1}$).

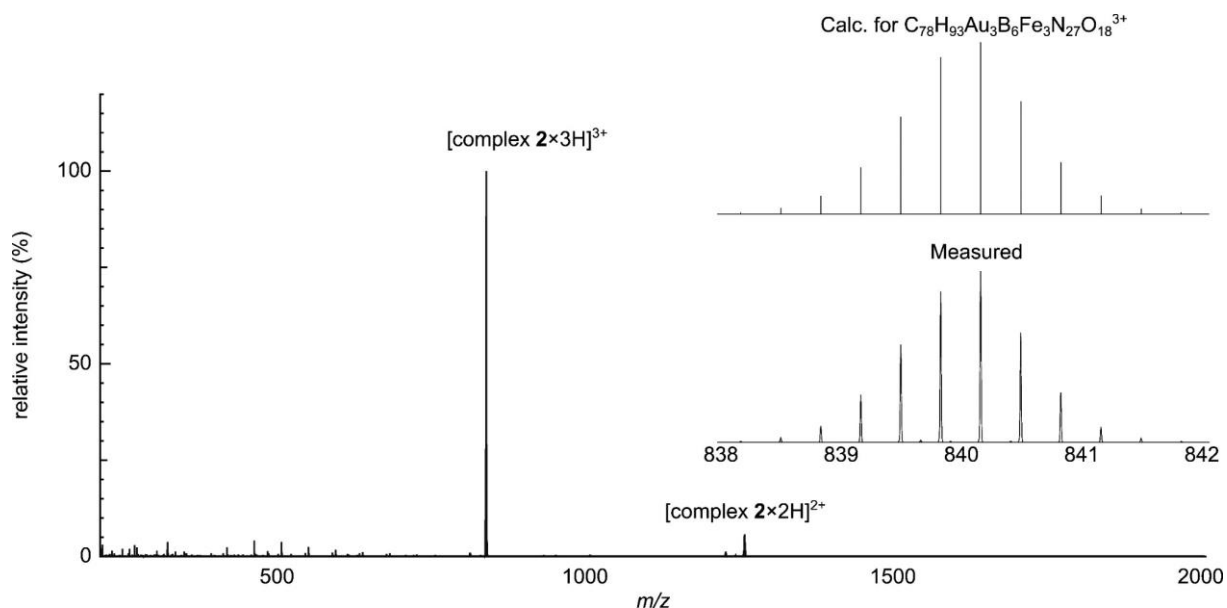
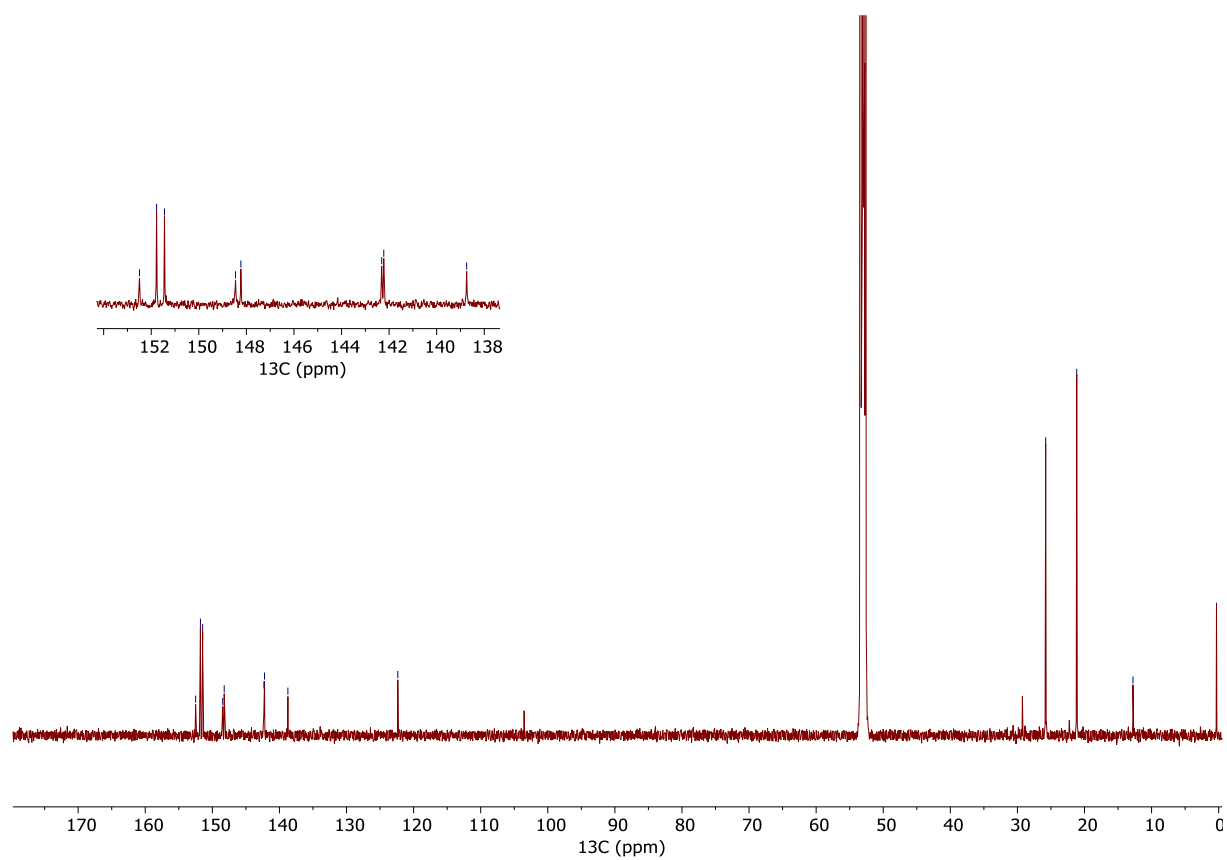
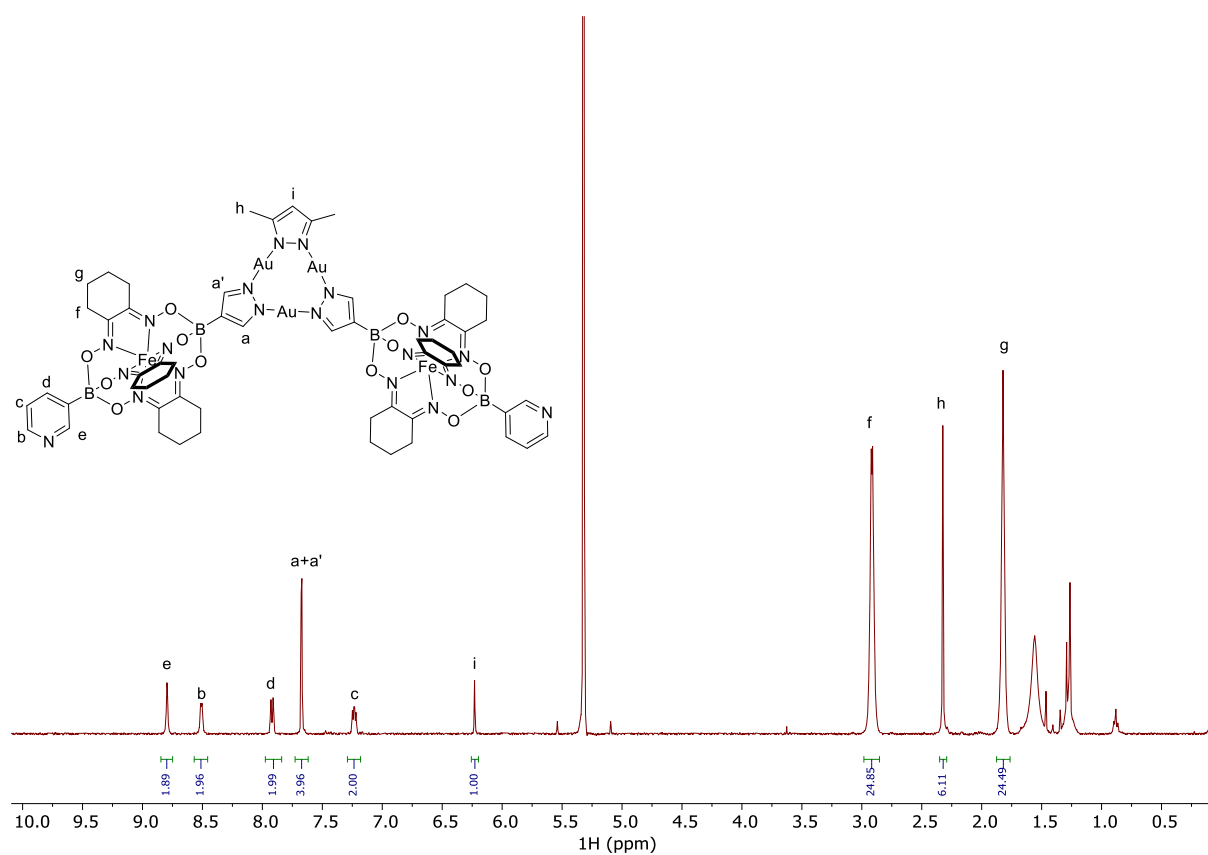


Figure S6. High-resolution ESI mass spectrum of complex **2**.

2.3 Synthesis of complex **3**

Triethylamine (30 μ L, 0.1 mmol) was added to a stirred solution of AuCl(SMe₂) (35.6 mg, 0.12 mmol), complex **1** (51 mg, 0.08 mmol), and 3,5-dimethylpyrazole (3.84 mg, 0.04 mmol) in THF (20 mL). The mixture was stirred for 18 h at RT under an atmosphere of N₂. The mixture was concentrated to about half the volume, and MeOH (approximately 30 mL) was added to precipitate the product and to dissolve the ammonium salt. The product was isolated by centrifugation, dissolved in CH₂Cl₂, and purified by column chromatography on silica with a mixture of CH₂Cl₂/MeOH (98:2) as eluent. Yield 12.3 mg, 15%. ¹H NMR (400 MHz, CD₂Cl₂) δ 8.80 (s, 2H), 8.51 (d, J = 4.9 Hz, 2H), 7.92 (d, J = 7.5 Hz, 2H), 7.73 – 7.62 (m, 4H), 7.27 – 7.20 (m, 2H), 6.23 (s, 1H), 2.94 – 2.89 (m, 24H), 2.32 (s, 6H), 1.86 – 1.79 (m, 24H). ¹³C NMR (151 MHz, CD₂Cl₂) δ 152.49, 151.77, 151.44, 148.46, 148.23, 142.31, 142.22, 138.74, 122.36, 103.95, 25.79, 21.16, 12.78. (C-B pyrazole and C-B pyridine not detected). ESI-MS: m/z calculated for C₅₇H₆₉Au₃B₄Fe₂N₂₀O₁₂²⁺ [M+2H]²⁺ 986.1730, found 986.1778.



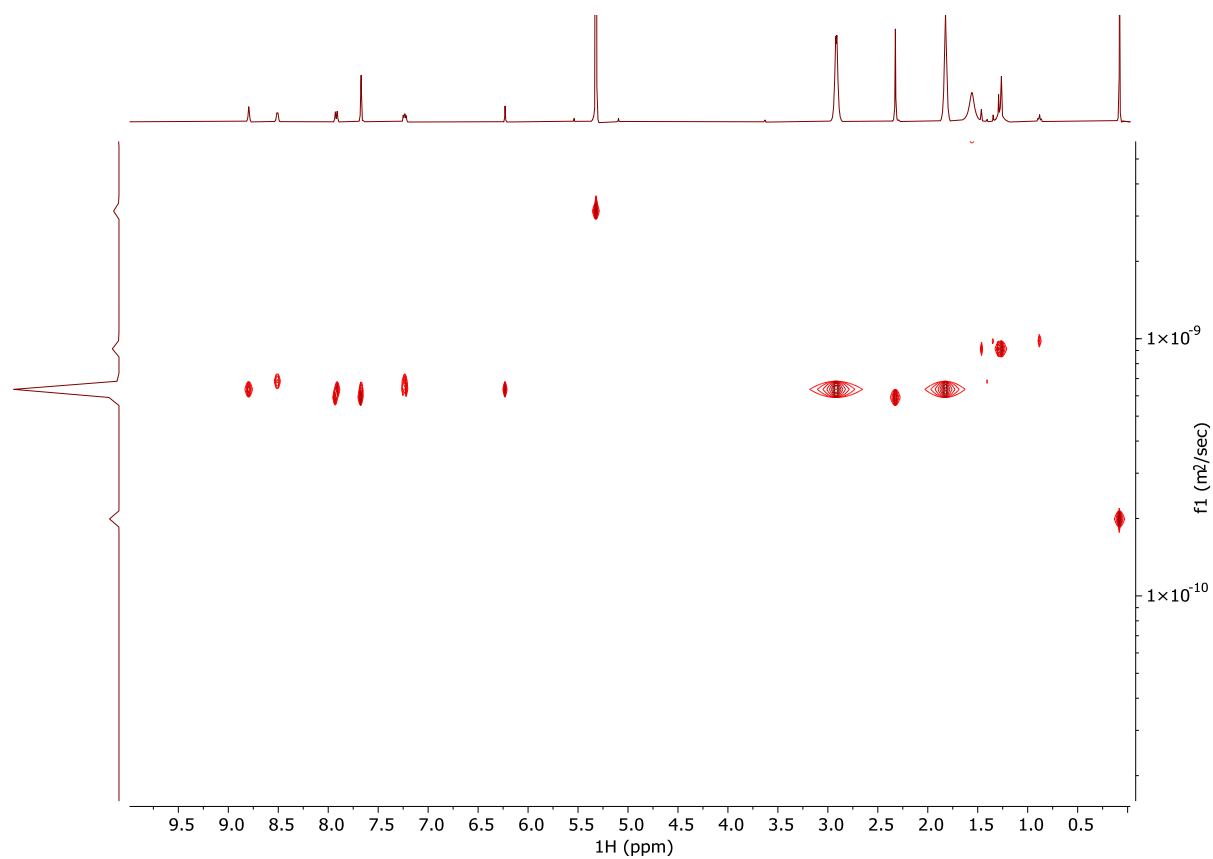


Figure S9. ^1H - ^1H DOSY NMR spectrum (400 MHz, CD_2Cl_2) of complex **3** ($D = 6.36 \times 10^{-10} \text{ m}^2\text{s}^{-1}$).

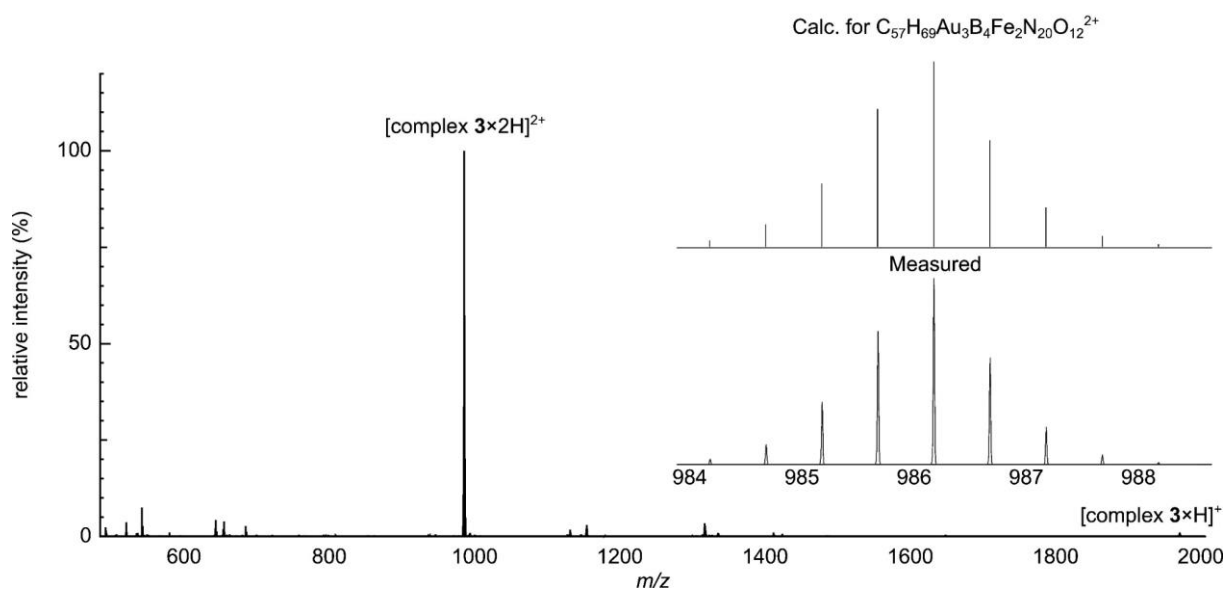


Figure S10. High-resolution ESI mass spectrum of complex **3**.

2.4 Synthesis of cage 4

A stock solution of $[\text{Pd}(\text{CH}_3\text{CN})_4](\text{BF}_4)_2$ (4.66 mg, 10.5 μmol) in $\text{DMSO-}d_6$ (349 μL) was prepared and 33 μL were added to a suspension of complex **2** (3.31 mg, 1.31 μmol) in $\text{DMSO-}d_6$ (477 μL). The mixture was heated to 60 $^\circ\text{C}$ for 15 h, during which a clear deep red solution was formed. ^1H NMR (400 MHz, $\text{DMSO-}d_6$, 298K) δ 8.67 (s, ~1H, $\text{H}_{b/d}$), 8.25 (s, ~1H, H_c), 7.82 – 7.38 (m, ~4H, $\text{H}_{b/d}$, H_a , and H_e). The broad signals compromise the accuracy of the integrals. The CH_2 protons of the Fe clathrochelate complex give rise to broad and ill-defined peaks between 1 and 3.5 ppm. Raising the temperature did not give better-resolved CH_2 signals.

^{13}C NMR: we have attempted to obtain a ^{13}C NMR spectrum of cage **4** using a 600 MHz instrument. Despite an acquisition time of several hours, we were not able to obtain a spectrum with acceptable signal-to-noise ratio.

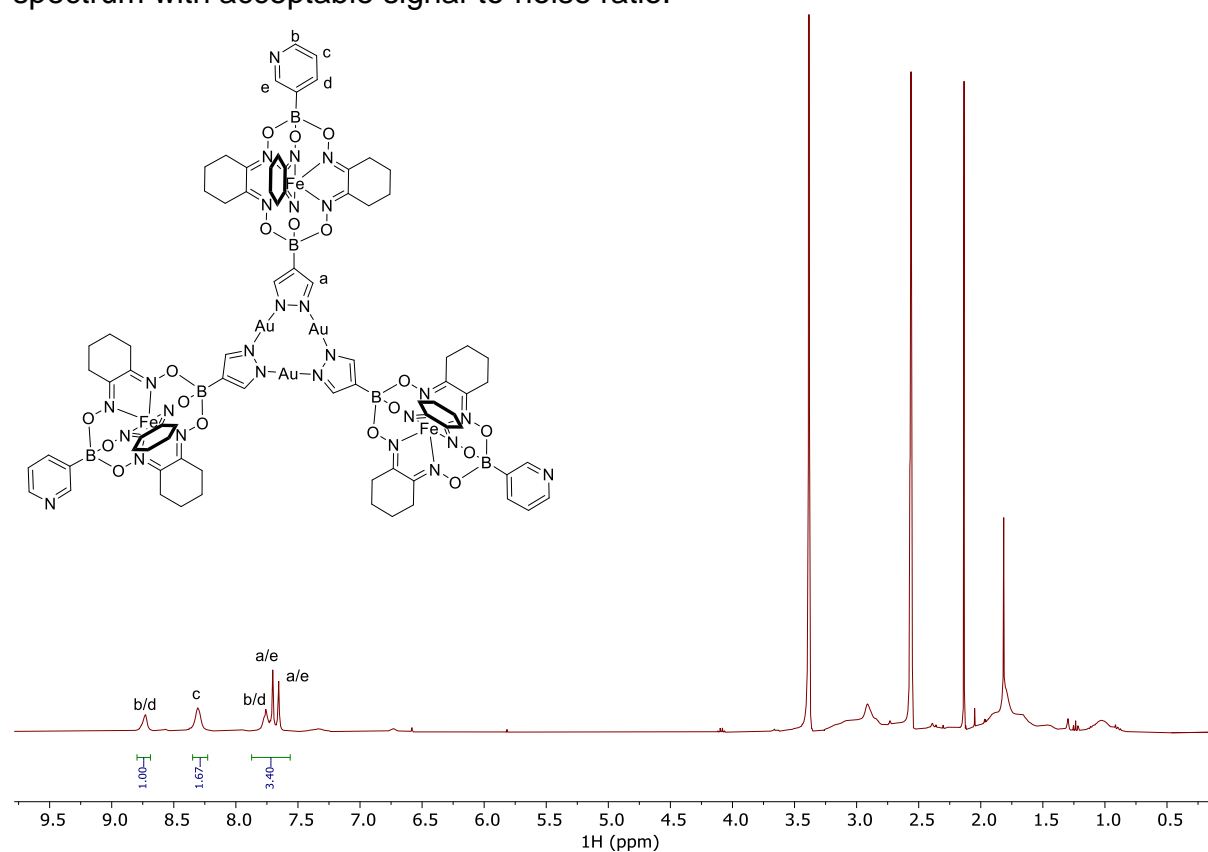


Figure S11. ^1H NMR spectrum (400 MHz, $\text{DMSO-}d_6$, 298 K) of cage **4**. The small, broad peaks at 6.6 and 7.3 ppm point to the presence of a minor side product.

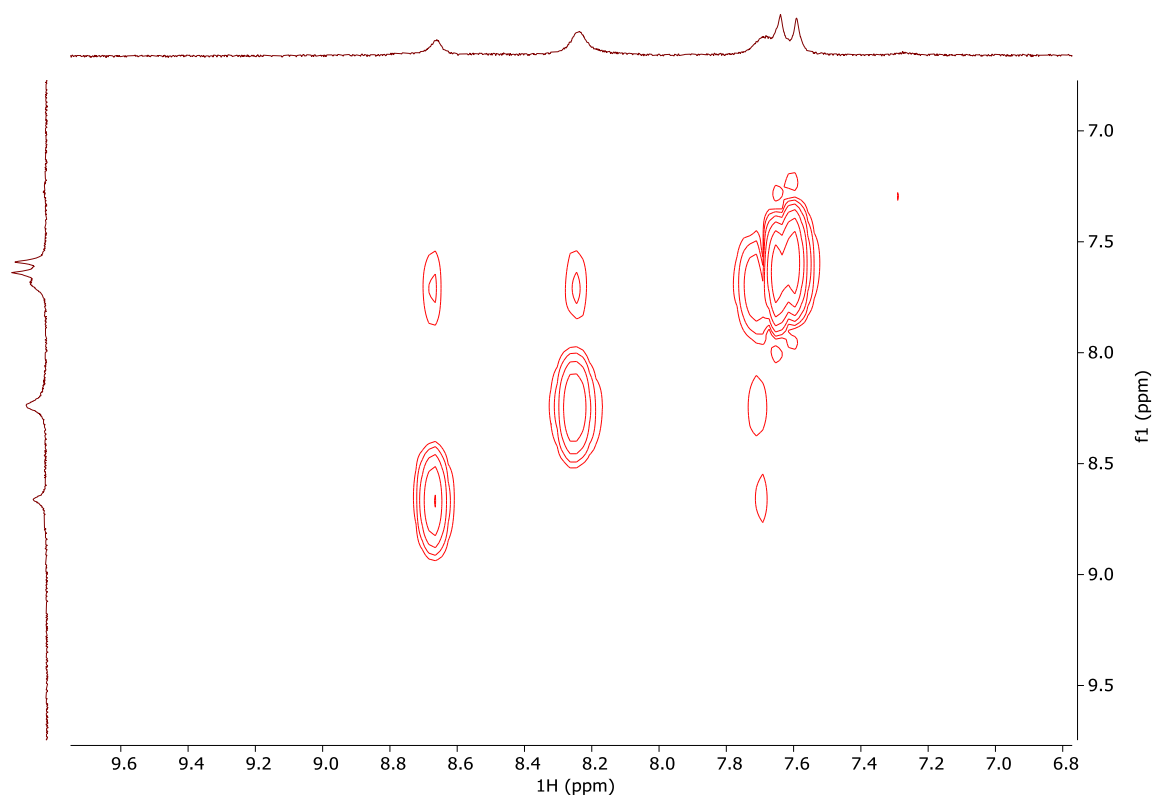


Figure S12. Aromatic part of ^1H - ^1H COSY NMR spectrum (400 MHz, CDCl_3 , 298 K) of cage 4.

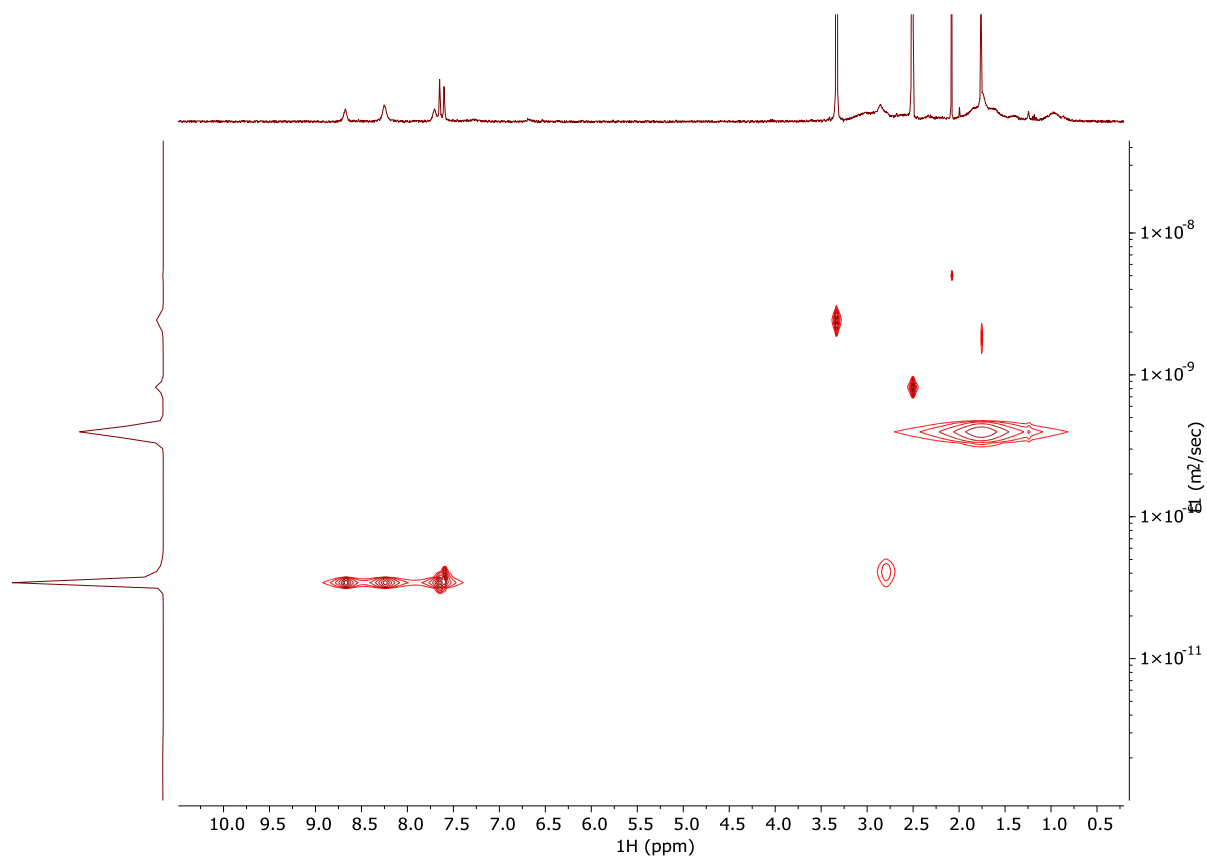


Figure S13. ^1H - ^1H DOSY NMR spectrum (400 MHz, $\text{DMSO}-d_6$) of cage 4 ($D = 3.51 \times 10^{-11} \text{ m}^2\text{s}^{-1}$, the calculated solvodynamic diameter of the cage is 4.3 nm).

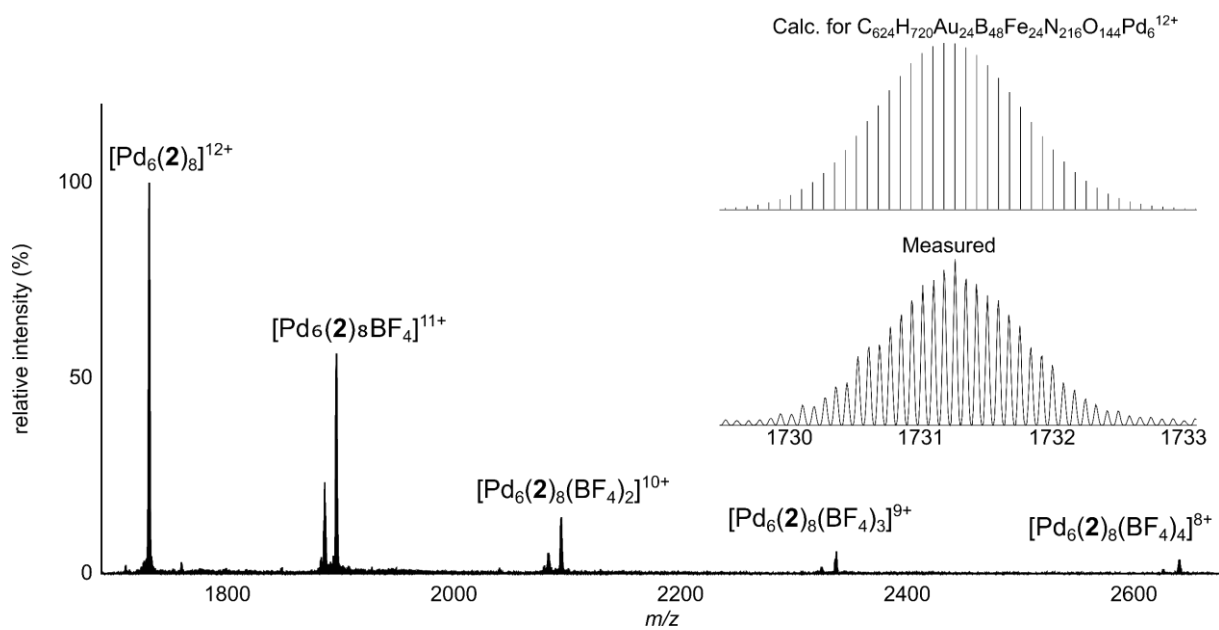


Figure S14. High-resolution ESI mass spectrum of cage **4**.

2.5 Synthesis of cage 5

Stock solution of $[\text{Pd}(\text{CH}_3\text{CN})_4](\text{BF}_4)_2$ (1.55 mg, 3.49 μmol) in $\text{DMSO-}d_6$ (90 μL) was prepared and 20 μL were added to a suspension of complex **3** (3.04 mg, 1.54 μmol) in $\text{DMSO-}d_6$ (480 μL). The mixture was heated to 60 $^\circ\text{C}$ while stirring. A clear deep red solution was observed within 15 min. The solution was kept at 60 $^\circ\text{C}$ overnight. ^1H NMR (400 MHz, $\text{DMSO-}d_6$, 333 K) δ 8.68 (d, $J = 5.7$ Hz, 2H), 8.29 (s, 2H), 8.24 (d, $J = 7.5$ Hz, 2H), 7.72 – 7.63 (m, 6H), 6.32 (s, 1H), 2.84 (s, 12H), 2.40 (s, 9H, theoretic 12H), 2.27 (s, 6H), 1.67 – 1.46 (m, 22H, theoretic 24H).

As in the case of cage **4**, we were not able to obtain a ^{13}C NMR spectrum of acceptable quality.

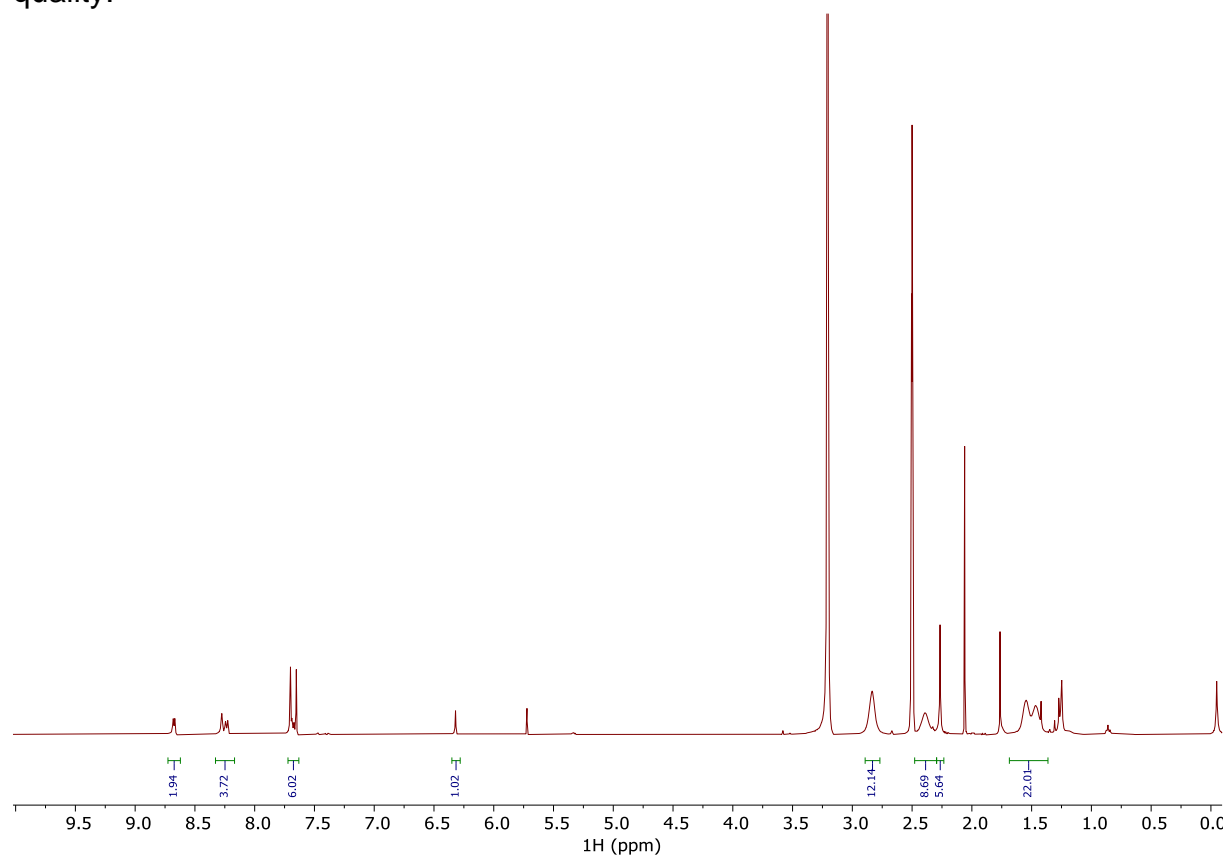


Figure S15. ^1H NMR spectrum (400 MHz, $\text{DMSO-}d_6$, 333 K) of cage **5**.

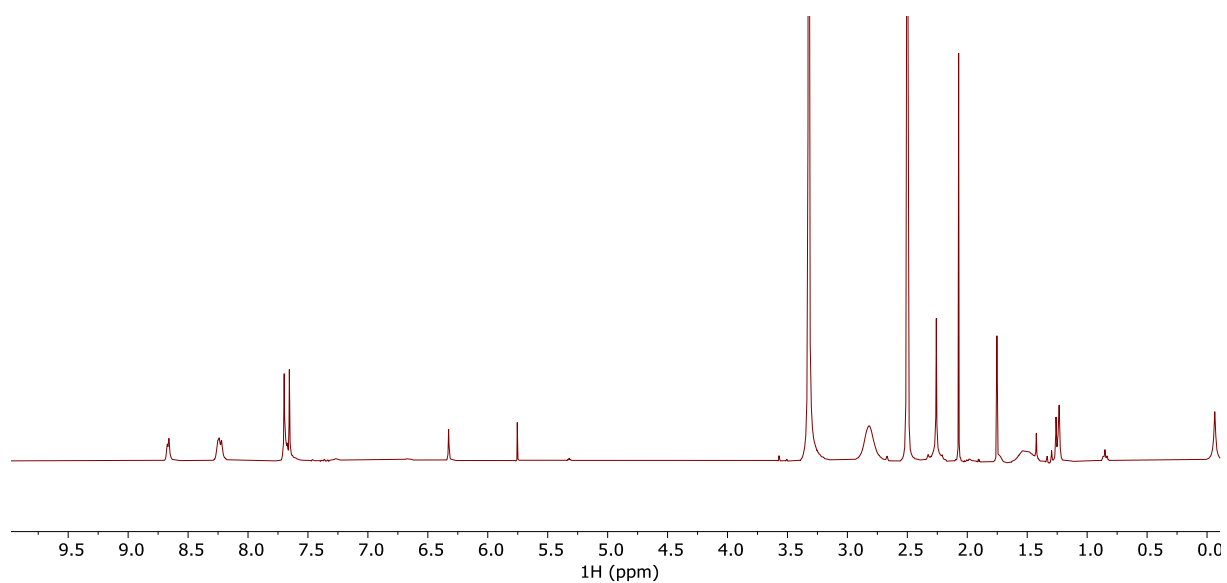


Figure S16. ^1H NMR spectrum (400 MHz, $\text{DMSO-}d_6$, 298 K) of cage **5**.

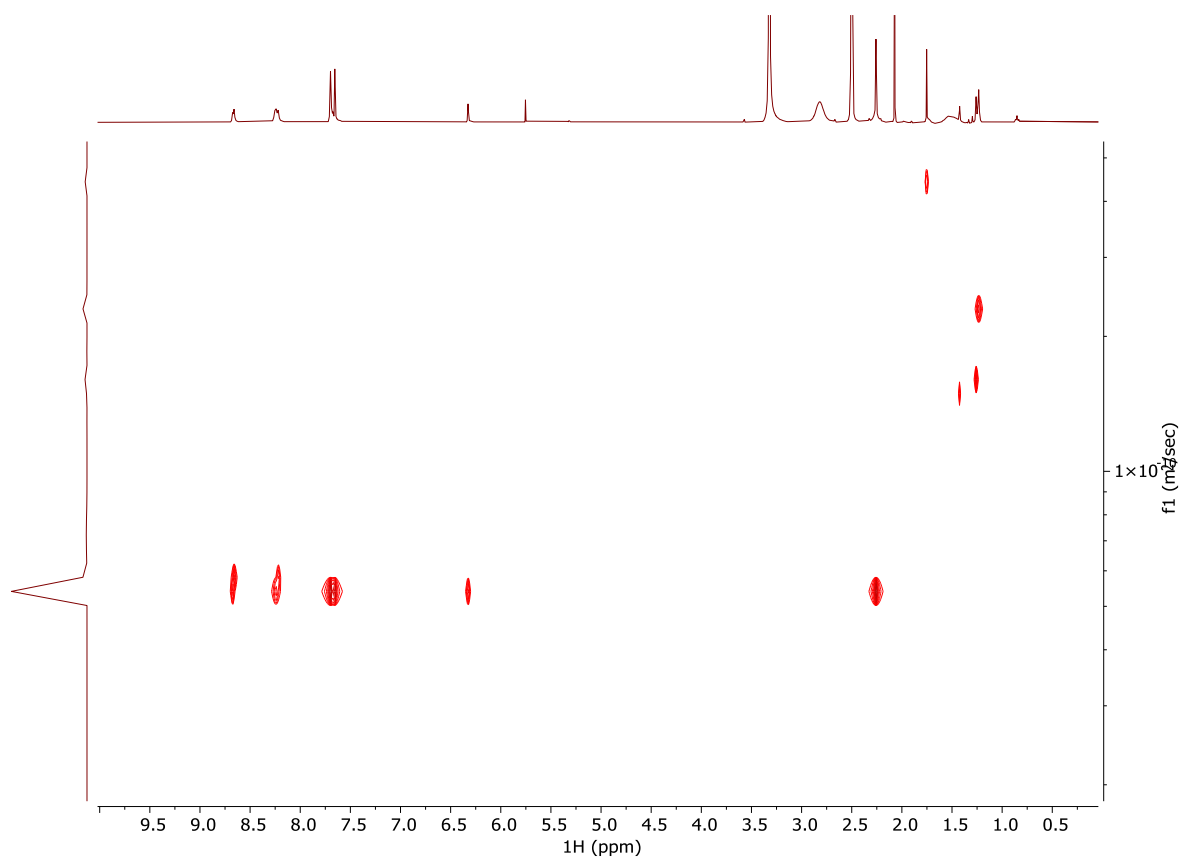


Figure S17. ^1H - ^1H DOSY NMR spectrum (400 MHz, $\text{DMSO-}d_6$) of cage **5** ($D = 5.41 \times 10^{-11} \text{ m}^2\text{s}^{-1}$, the calculated solvodynamic diameter of the cage is 2.8 nm).

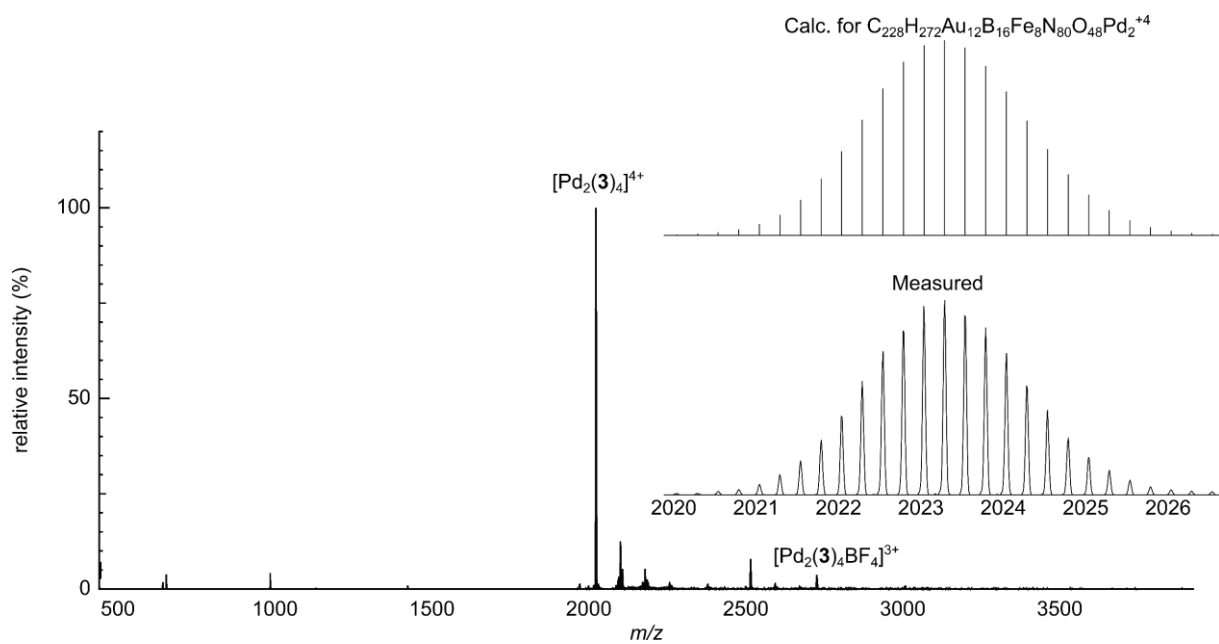


Figure S18. High-resolution ESI mass spectrum of cage **5**.

3 Crystallographic Data

3.1 Crystal structure of complex 1

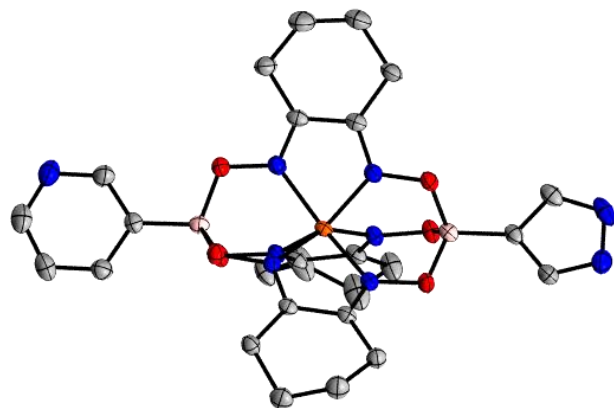


Figure S19. ORTP plot at 50% probability of **1**.

Crystals suitable for SCXRD analysis were obtained by slow evaporation of a concentrated MeOH solution of **1**.

A clear intense orange plate-shaped crystal with dimensions $0.50 \times 0.35 \times 0.03 \text{ mm}^3$ was mounted. Data were collected using an XtaLAB Synergy R, DW system, HyPix-Arc 150 diffractometer operating at $T = 140.00(10) \text{ K}$. The structure was solved with the *ShelXT*⁶ solution program using dual methods and by using *Olex2*⁷ as the graphical interface. The model was refined with *ShelXL*⁸ using full matrix least squares minimisation on F^2 .

Data were measured using ω scans with $\text{CuK}\alpha$ radiation. The diffraction pattern was indexed and the total number of runs and images was based on the strategy calculation from the program *CrysAlisPro*.⁹ The maximum resolution achieved was $\theta = 75.235^\circ$ (0.80 Å).

The unit cell was refined using *CrysAlisPro* on 17269 reflections, 36% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using *CrysAlisPro*. The final completeness is 99.70 % out to 75.235° in θ . A Gaussian absorption correction was performed using *CrysAlisPro*. Numerical absorption correction based on Gaussian integration over a multifaceted crystal model. Empirical absorption correction using spherical harmonics as implemented in SCALE3 ABSPACK scaling algorithm. The absorption coefficient μ of this material is 4.301 mm^{-1} at this wavelength ($\lambda = 1.54184 \text{ \AA}$) and the minimum and maximum transmissions are 0.063 and 1.000.

The structure was solved in the space group $P\bar{1}$ (# 2) determined by the *ShelXT* structure solution program using dual methods and refined by full matrix least squares minimisation on R^2 using version 2019/3 of *ShelXL*. All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model, but the hydrogen atoms bound to O13 were refined freely.

The value of Z is 2. This means that there are two independent molecules in the asymmetric unit. The moiety formula is $C_{26}H_{30}B_2FeN_9O_6$, H_2O , 1.5 (CH_3OH) .

Table S1. Crystal data and structure refinement for ligand **1**

Formula	C _{27.65} H _{39.3} B ₂ FeN ₉ O _{8.5}
<i>D</i> _{calc.} / g cm ⁻³	1.461
μ /mm ⁻¹	4.297
Formula Weight	711.25
Colour	clear intense orange
Shape	plate-shaped
Size/mm ³	0.50×0.35×0.03
<i>T</i> /K	140.00(10)
Crystal System	triclinic
Space Group	<i>P</i> $\bar{1}$
<i>a</i> /Å	10.4293(2)
<i>b</i> /Å	14.3513(2)
<i>c</i> /Å	22.8201(4)
α /°	105.9779(14)
β /°	90.6964(15)
γ /°	99.3977(14)
<i>V</i> /Å ³	3233.67(10)
<i>Z</i>	4
<i>Z</i> '	2
Wavelength/Å	1.54184
Radiation type	CuK α
θ _{min} /°	3.252
θ _{max} /°	75.235
Measured Refl's.	47351
Indep't Refl's	12716
Refl's $I \geq 2\sigma(I)$	10382
<i>R</i> _{int}	0.0399
Parameters	924
Restraints	38
Largest Peak/e Å ⁻³	0.636
Deepest Hole/e Å ⁻³	-0.444
GooF	1.064
<i>wR</i> ₂ (all data)	0.1429
<i>wR</i> ₂	0.1362
<i>R</i> ₁ (all data)	0.0618
<i>R</i> ₁	0.0498
CCDC number	2325914

3.2 Crystal structure of complex 2

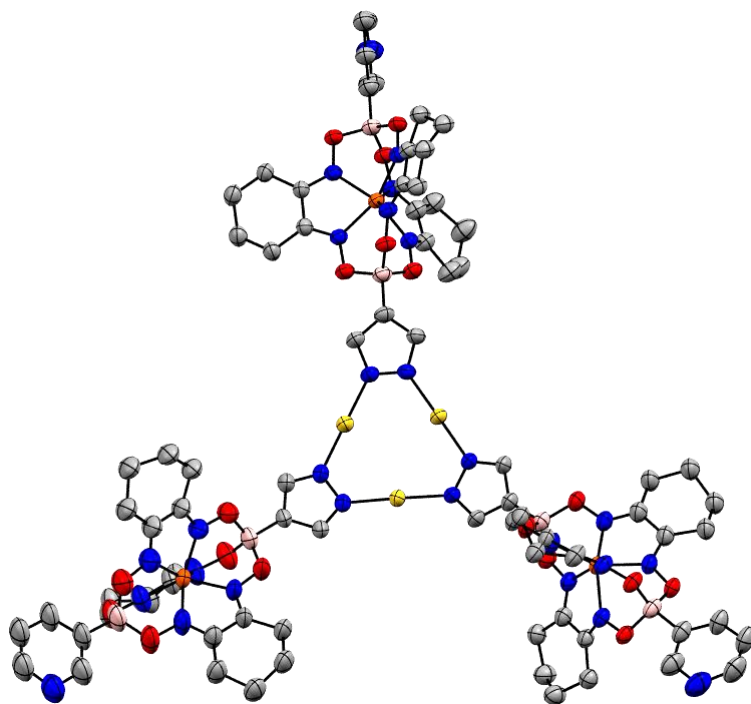


Figure S20. ORTP plot at 50% probability of **2**.

Crystals suitable for SCXRD analysis were obtained slow evaporation of dilute solution of complex **2** in CHCl_3 : acetonitrile.

A clear intense orange plate-shaped crystal with dimensions $0.22 \times 0.06 \times 0.03 \text{ mm}^3$ was mounted. Data were collected using a SuperNova, Dual, Cu at home/near, Atlas diffractometer operating at $T = 230.00(10) \text{ K}$. The structure was solved with the *ShelXT* solution program using dual methods and by using *Olex2* as the graphical interface. The model was refined with *ShelXL* using full matrix least squares minimisation on F^2 .

Data were measured using ω scans with $\text{CuK}\alpha$ radiation. The diffraction pattern was indexed and the total number of runs and images was based on the strategy calculation from the program *CrysAlis^{Pro}*. The maximum resolution achieved was $\theta = 77.040^\circ$ (0.79 Å).

The unit cell was refined using *CrysAlis^{Pro}* on 12566 reflections, 24% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using *CrysAlis^{Pro}*. The final completeness is 99.80 % out to 77.040° in θ . A Gaussian absorption correction was performed using *CrysAlis^{Pro}*. Numerical absorption correction based on Gaussian integration over a multifaceted crystal model. Empirical absorption correction using spherical harmonics as implemented in SCALE3 ABSPACK scaling

algorithm. The absorption coefficient μ of this material is 13.097 mm^{-1} at this wavelength ($\lambda = 1.54184 \text{ \AA}$) and the minimum and maximum transmissions are 0.278 and 0.857.

The structure was solved in the space group $P\bar{1}$ (# 2) determined by the *ShelXT* structure solution program using dual methods and refined by full matrix least squares minimisation on F^2 using version 2019/3 of *ShelXL*. All non-hydrogen atoms were refined anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model.

There is a single formula unit in the asymmetric unit, which is represented by the reported sum formula. In other words: Z is 2 and Z' is 1. The moiety formula is $\text{C}_{78}\text{H}_{90}\text{Au}_3\text{B}_6\text{Fe}_3\text{N}_{27}\text{O}_{18}, \text{CHCl}_3, \text{C}_2\text{H}_3\text{N}, 0.85[\text{CHCl}_3]$.

A solvent mask was calculated, and 97 electrons were found in a volume of 599 \AA^3 in two voids per unit cell. This is consistent with the presence of 0.85 solvent molecule of Chloroform per Asymmetric Unit which accounts for 99 electrons per unit cell.

Table S2. Crystal data and structure refinement for complex **2**

Formula	C _{81.85} H _{94.85} Au ₃ B ₆ Cl _{5.55} Fe ₃ N ₂₈ O ₁₈
$D_{calc.}/\text{g cm}^{-3}$	1.792
μ/mm^{-1}	13.097
Formula Weight	2778.96
Colour	clear intense orange
Shape	plate-shaped
Size/ mm^3	0.22×0.06×0.03
T/K	230.00(10)
Crystal System	triclinic
Space Group	$P\bar{1}$
$a/\text{\AA}$	9.9930(3)
$b/\text{\AA}$	21.4450(7)
$c/\text{\AA}$	26.8363(8)
α°	108.765(3)
β°	99.440(3)
γ°	102.247(3)
$V/\text{\AA}^3$	5151.0(3)
Z	2
Z'	1
Wavelength/ \AA	1.54184
Radiation type	CuK α
θ_{min}°	3.376
θ_{max}°	77.040
Measured Refl's.	51716
Indep't Refl's	21181
Refl's $I \geq 2\sigma(I)$	14173
R_{int}	0.0607
Parameters	1355
Restraints	1104
Largest Peak/ $e \text{\AA}^{-3}$	1.936
Deepest Hole/ $e \text{\AA}^{-3}$	-1.424
Goof	1.009
wR_2 (all data)	0.1429
wR_2	0.1242
R_1 (all data)	0.0837
R_1	0.0513
CCDC number	2325913

3.3 Crystal structure of complex 3

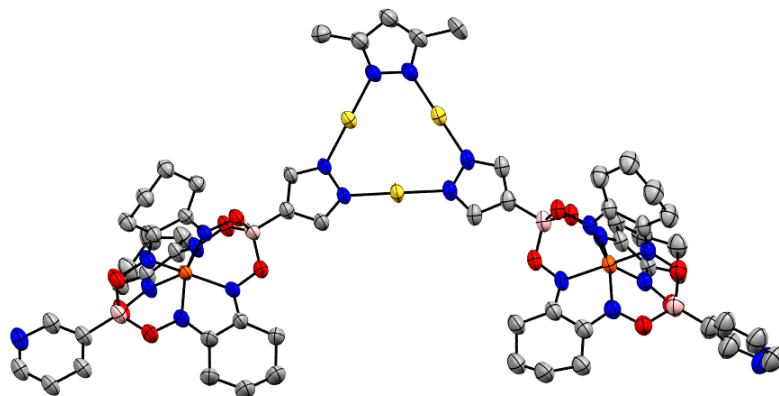


Figure S21. ORTP plot at 50% probability of **3**.

Crystals suitable for SCXRD analysis were obtained by layering acetonitrile onto a solution of complex **3** in CH_2Cl_2 .

A clear intense orange plate-shaped crystal with dimensions $0.14 \times 0.08 \times 0.03 \text{ mm}^3$ was mounted. Data were collected using an XtaLAB Synergy R, DW system, HyPix-Arc 150 diffractometer operating at $T = 140.00(10) \text{ K}$. The structure was solved with the *ShelXT* solution program using dual methods and by using *Olex2* as the graphical interface. The model was refined with *ShelXL* using full matrix least squares minimisation on R^2 .

Data were measured using ω scans with $\text{CuK}\alpha$ radiation. The diffraction pattern was indexed and the total number of runs and images was based on the strategy calculation from the program *CrysAlisPro* system. The maximum resolution achieved was $\theta = 76.311^\circ$ (0.79 \AA).

The unit cell was refined using *CrysAlisPro* on 28377 reflections, 155% of the observed reflections.

Data reduction, scaling and absorption corrections were performed using *CrysAlisPro*. The final completeness is 99.90 % out to 76.311° in Q . A Gaussian absorption correction was performed using *CrysAlisPro*. Numerical absorption correction based on Gaussian integration over a multifaceted crystal model. Empirical absorption correction using spherical harmonics as implemented in SCALE3 ABSPACK scaling algorithm. The absorption coefficient μ of this material is 15.683 mm^{-1} at this wavelength ($\lambda = 1.54184 \text{ \AA}$) and the minimum and maximum transmissions are 0.220 and 0.803.

The structure was solved in the space group $P\bar{1}$ (# 2) by the *ShelXT* structure solution program using dual methods and refined by full matrix least squares minimisation on R^2 using version 2019/3 of *ShelXL*. All non-hydrogen atoms were refined

anisotropically. Hydrogen atom positions were calculated geometrically and refined using the riding model.

_refine_special_details: Refined as a 2-component twin.

_twin_special_details: Component 2 rotated by 2.4718° around [0.70 0.44 -0.56] (reciprocal) or [0.81 0.51 -0.29] (direct)

The value of Z' is 0.5. This means that only half of the formula unit is present in the asymmetric unit, with the other half consisting of symmetry equivalent atoms. The moiety formula is C₁₁₄H₁₃₄Au₆B₈Fe₄N₄₀O₂₄, 2CH₂Cl₂, 5[CH₂Cl₂].

_platon_squeeze_special_details: A solvent mask was calculated, and 233 electrons were found in a volume of 824 Å³ in one void per unit cell. This is consistent with the presence of five solvent molecule of DCM per Asymmetric Unit which account for 210 electrons per unit cell.

Table S3. Crystal data and structure refinement for complex **3**

Formula	C ₁₂₁ H ₁₄₈ Au ₆ B ₈ Cl ₁₄ Fe ₄ N ₄₀ O ₂₄
<i>D</i> _{calc.} / g cm ⁻³	1.886
<i>μ</i> /mm ⁻¹	15.683
Formula Weight	4534.76
Colour	clear intense orange
Shape	plate-shaped
Size/mm ³	0.14×0.08×0.03
<i>T</i> /K	140.00(10)
Crystal System	triclinic
Space Group	<i>P</i> $\bar{1}$
<i>a</i> /Å	13.03697(15)
<i>b</i> /Å	13.61355(15)
<i>c</i> /Å	23.2896(3)
<i>α</i> /°	77.3494(11)
<i>β</i> /°	88.5561(10)
<i>γ</i> /°	81.9706(10)
<i>V</i> /Å ³	3993.48(9)
<i>Z</i>	1
<i>Z</i> '	0.5
Wavelength/Å	1.54184
Radiation type	CuKα
<i>θ</i> _{min} /°	1.944
<i>θ</i> _{max} /°	76.311
Measured Refl's.	18352
Indep't Refl's	18352
Refl's I ≥ 2σ(I)	14104
<i>R</i> _{int}	n/a
Parameters	913
Restraints	1726
Largest Peak/e Å ⁻³	1.752
Deepest Hole/e Å ⁻³	-1.650
Goof	1.050
<i>wR</i> ₂ (all data)	0.1495
<i>wR</i> ₂	0.1377
<i>R</i> ₁ (all data)	0.0731
<i>R</i> ₁	0.0543
CCDC number	2325915

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