

S1

The Facile Assembly of Bis-, Tris- and Poly(triazaphosphole) Systems Using "Click"

Chemistry

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SUPPLEMENTARY INFORMATION

Table S1. Summary of crystallographic data for **3**, **4** and [Fe{C₅H₄(N₃PCBu^tH)}₂][CF₃SO₃]₂ **1S**.

	3	4	1S
empirical formula	C ₂₀ H ₂₆ FeN ₆ P ₂	C ₁₄ H ₁₄ FeN ₆ P ₂	C ₂₂ H ₂₈ F ₆ FeN ₆ O ₆ P ₂ S ₂
formula weight	468.26	384.10	768.41
crystal system	monoclinic	monoclinic	triclinic
space group	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> 2 ₁ / <i>c</i>	<i>P</i> -1
<i>T</i> (K)	123(2)	123(2)	123(2)
<i>a</i> (Å)	17.573(4)	9.1840(18)	6.5273(1)
<i>b</i> (Å)	10.129(2)	13.517(3)	7.2151(1)
<i>c</i> (Å)	12.835(3)	12.433(3)	16.4648(3)
α (deg.)	90	90	97.988(2)
β (deg.)	93.64(3)	91.27(3)	90.106(2)
γ (deg.)	90	90	96.513(2)
vol (Å ³)	2280.1(8)	1543.1(5)	762.81(2)
<i>Z</i>	4	4	1
ρ (calcd) (g.cm ⁻³)	1.364	1.653	1.673
μ (mm ⁻¹)	0.820	1.192	0.821
<i>F</i> (000)	976	784	392
reflections collected	9466	5131	14897
unique reflections	4970	2722	4351
<i>R</i> _{int}	0.0198	0.0323	0.0454
R1 indices [<i>I</i> >2 σ (<i>I</i>)]	0.0313	0.0350	0.0389
wR2 indices (all data)	0.0777	0.0800	0.0977
Largest peak and hole (e.Å ⁻³)	0.37, -0.32	0.44, -0.26	0.44, -1.05
CCDC No.	925611	925612	925610

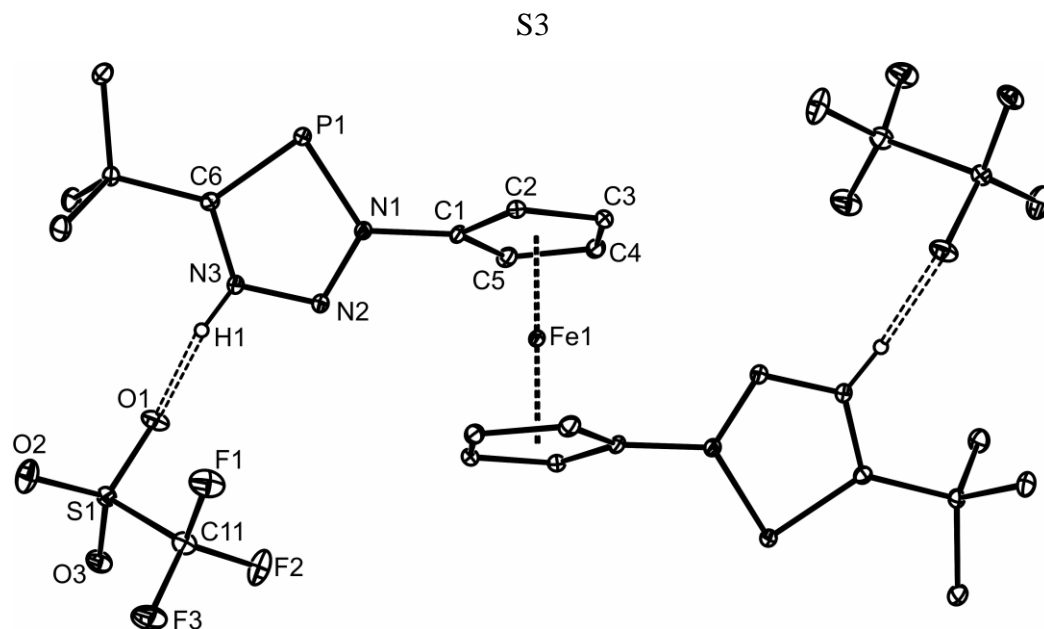


Figure S1. Molecular structure of **1S** (25% displacement ellipsoids are shown; hydrogens, except H(1), omitted). Selected bond lengths (Å) and angles (°): P(1)-N(1) 1.7036(16), P(1)-C(6) 1.7114(19), N(1)-N(2) 1.317(2), N(1)-C(1) 1.436(2), N(2)-N(3) 1.312(2), N(3)-C(6) 1.346(2), N(3)-H(1) 0.89(3), H(1)-O(1) 1.78(2), N(1)-P(1)-C(6) 86.27(8), N(3)-C(6)-P(1) 110.15(13), N(3)-H(1)-O(1) 163(1).

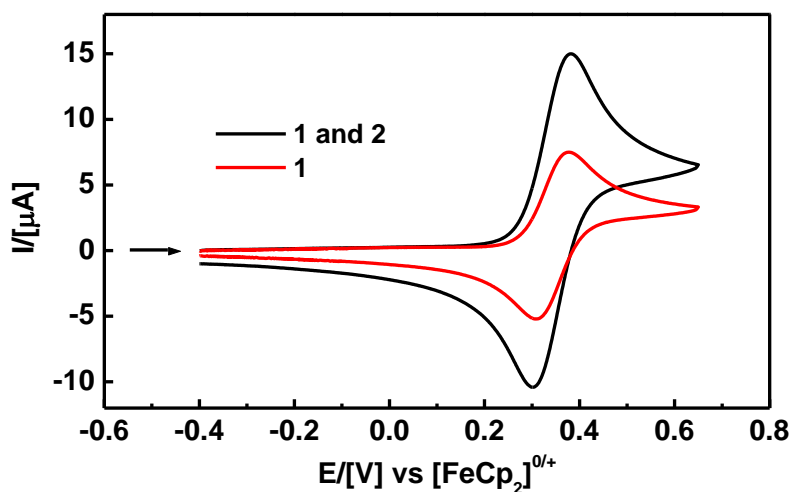


Figure S2: Cyclic voltammograms obtained at scan rate of 0.1 V s^{-1} with a 1.5 mm diameter glassy carbon disk electrode for 2 mM **3** (red curve) and 2 mM of both **3** and **4** (black curve) in CH_2Cl_2 (0.1 M $[\text{Bu}_4\text{N}][\text{PF}_6]$).

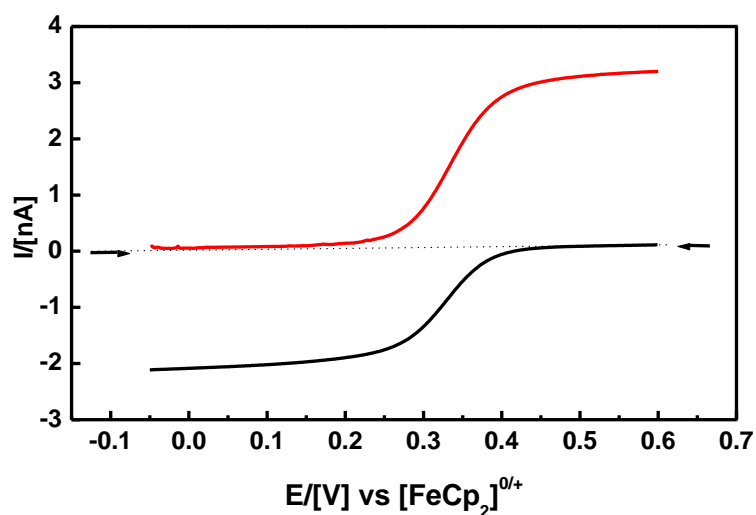


Figure S3: Near steady-state linear sweep voltammograms obtained at scan rate of 0.1 V s^{-1} with a Pt-microelectrode (10 μm diameter) before (red curve) and after (black curve) oxidative bulk electrolysis at $E_{\text{appl}} = 0.6 \text{ V}$ for 1.8 mM **3** in CH_2Cl_2 (0.1 M $[\text{Bu}_4\text{N}][\text{PF}_6]$).