

Figure S1. IR-Spectra of **1**

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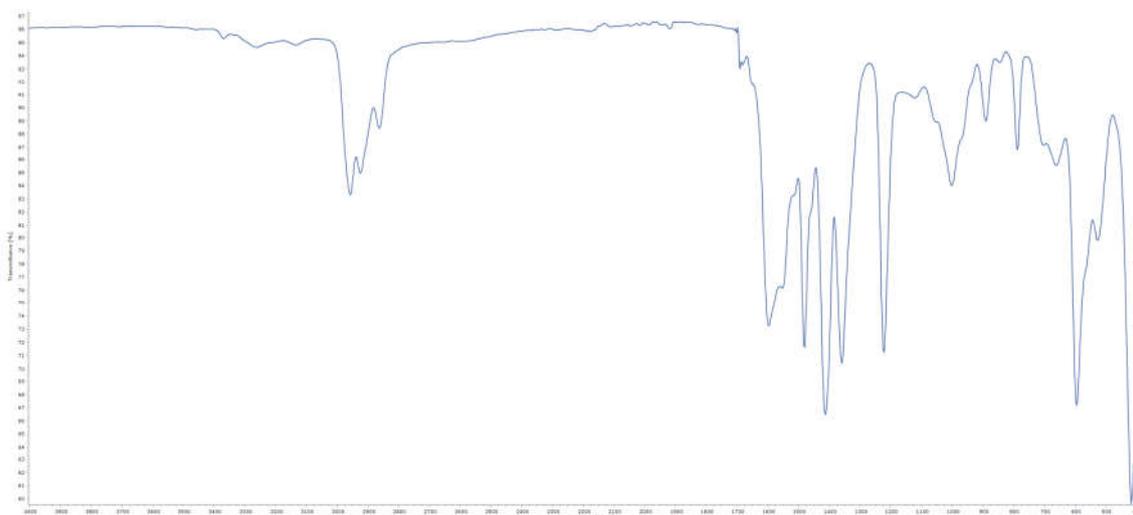


Figure S2. IR-Spectra of **2**

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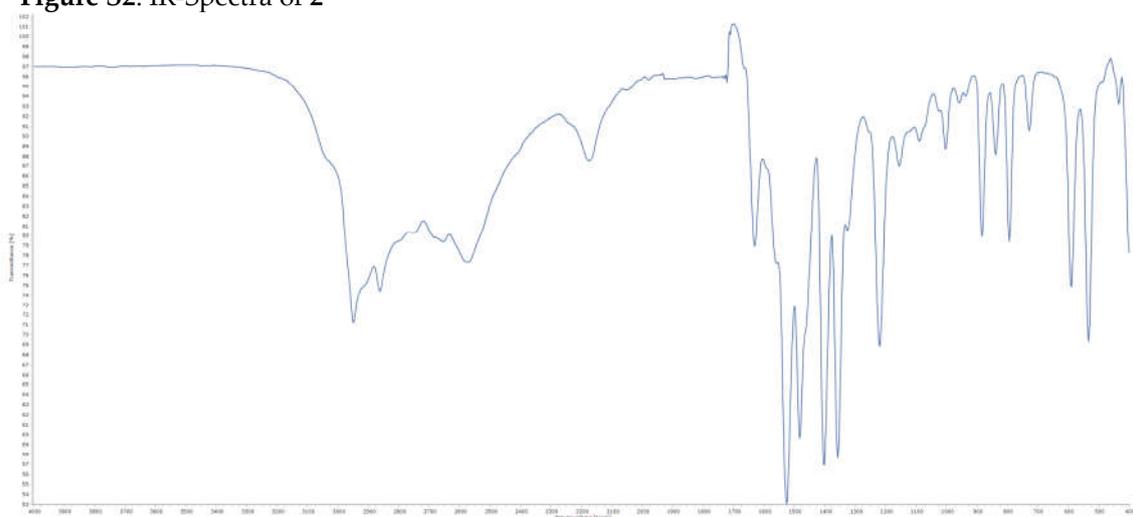


Figure S3 IR-Spectra of **3**

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To the iron(II) pivalate prepared in situ from $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (0.278 g, 1.0 mmol) and Kpiv (0.280 g, 2.0 mmol), 10 ml of acetonitrile was condensed and added 1,10-phenanthroline monohydrate (0.198 g, 1 mmol)

The filtrate was sealed in a glass ampoule, heated in an oil bath at a temperature of 120°C for 10 hours, further cooling to room temperature led to the formation of blue-green crystals



In air, blue-green crystals turn orange when exposed to acetone. However, powder in air without the action of a solvent is resistant to atmospheric oxygen for more than two months (see Figure S6)

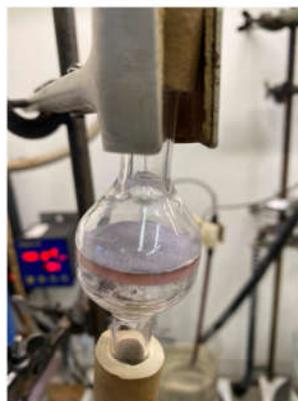


Isolated single crystals in a sealed glass ampoules

Figure S4. Synthesis of compound $[\text{Fe}_2(\text{piv})_4(\text{phen})_2] \mathbf{1}$



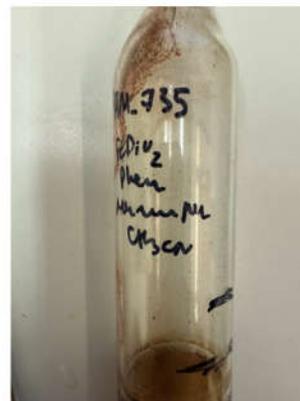
To the iron(II) pivalate prepared *in situ* from $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (0.278 g, 1.0 mmol) and Kpiv (0.280 g, 2.0 mmol), 10 ml of acetonitrile was condensed and added 1,10-phenanthroline monohydrate (0.198 g, 1 mmol). The red-colored reaction mixture was filtered from potassium sulfate.



A solution of 1,6-diaminohexane (0.130 g, 1.12 mmol) in 2 ml of anhydrous CH_3CN was added to 1 mmol of complex **1** prepared *in situ* in 10 ml of acetonitrile. The reaction mixture was sealed in a glass ampoule and heated in an oil bath at 100°C for 5 hours until a solution was formed (*caution! high pressure*)



Cooling to room temperature (24°C) led to the formation of red (**2**, 0.122 g, 38% per 1 mmol **1**) and white crystals ($(\text{piv})_2(\text{H}_2\text{hmda})$, **3**).



Isolated single crystals in a sealed glass ampoule. Separation of **2** and **3** was carried out by multiple decantation

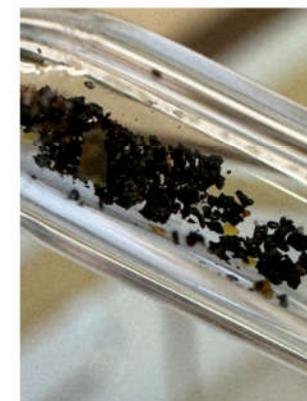


Figure S5. Synthesis of compound $[\text{Fe}_3\text{O}(\text{Piv})_6(\text{NH}_2(\text{CH}_2)_6\text{NH}_2)_{1.5}]_n$ (**2**) + **3**

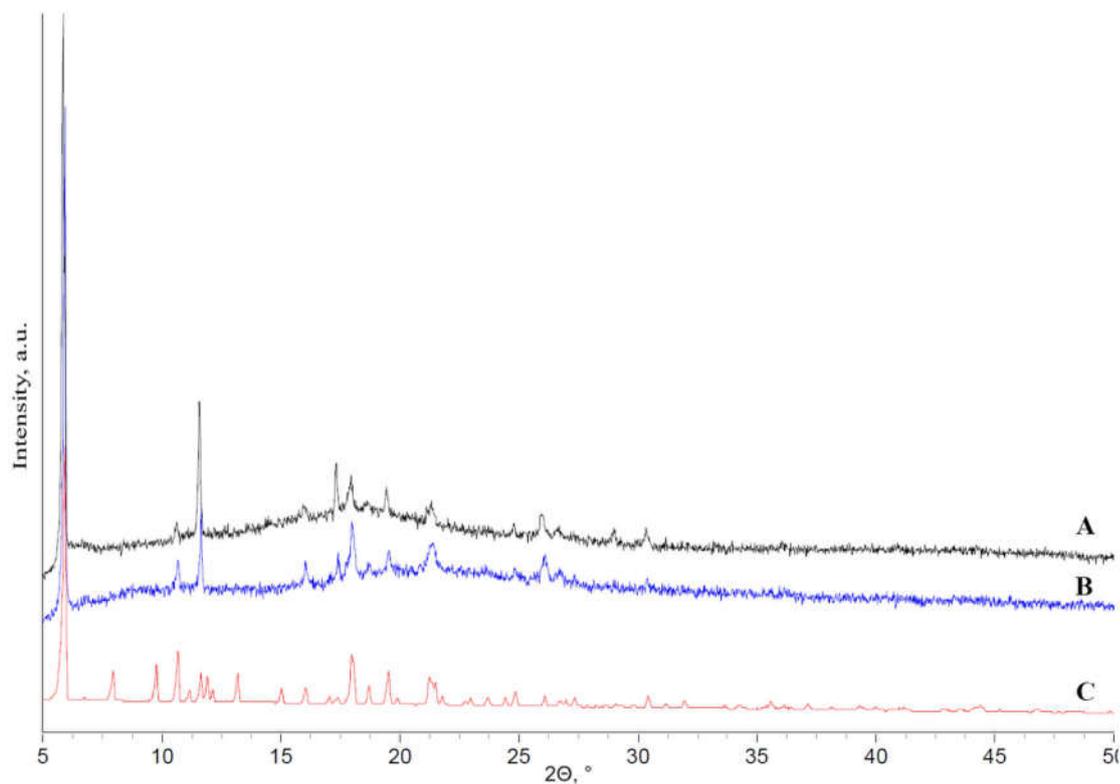


Figure S6. Comparison of experimental (A, black line 26 March 2024; B, blue line, 12 January 2024) and theoretical (C, red line) diffraction patterns for sample 1

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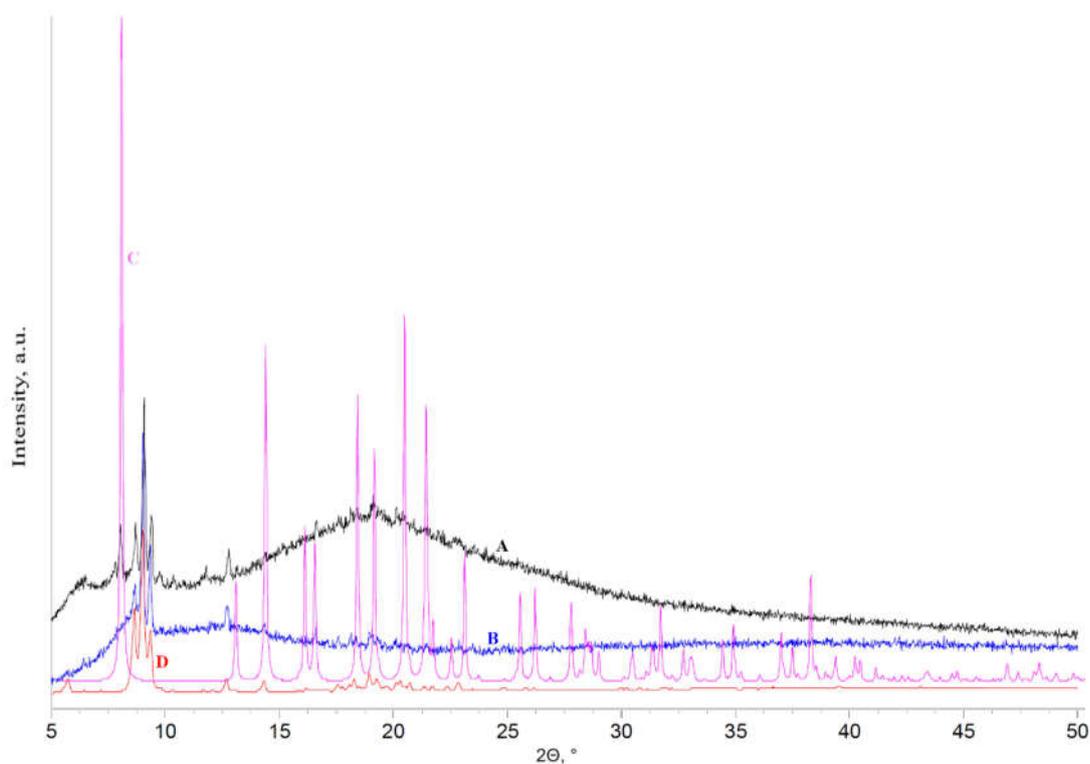


Figure S7. Comparison of experimental for sample 2 in time in air atmosphere (blue line (B) - after 40 min in air; black line (A) - after 13 h in air) and theoretical diffraction patterns for 2 (red line (C)) and 3 (pink line (D)).

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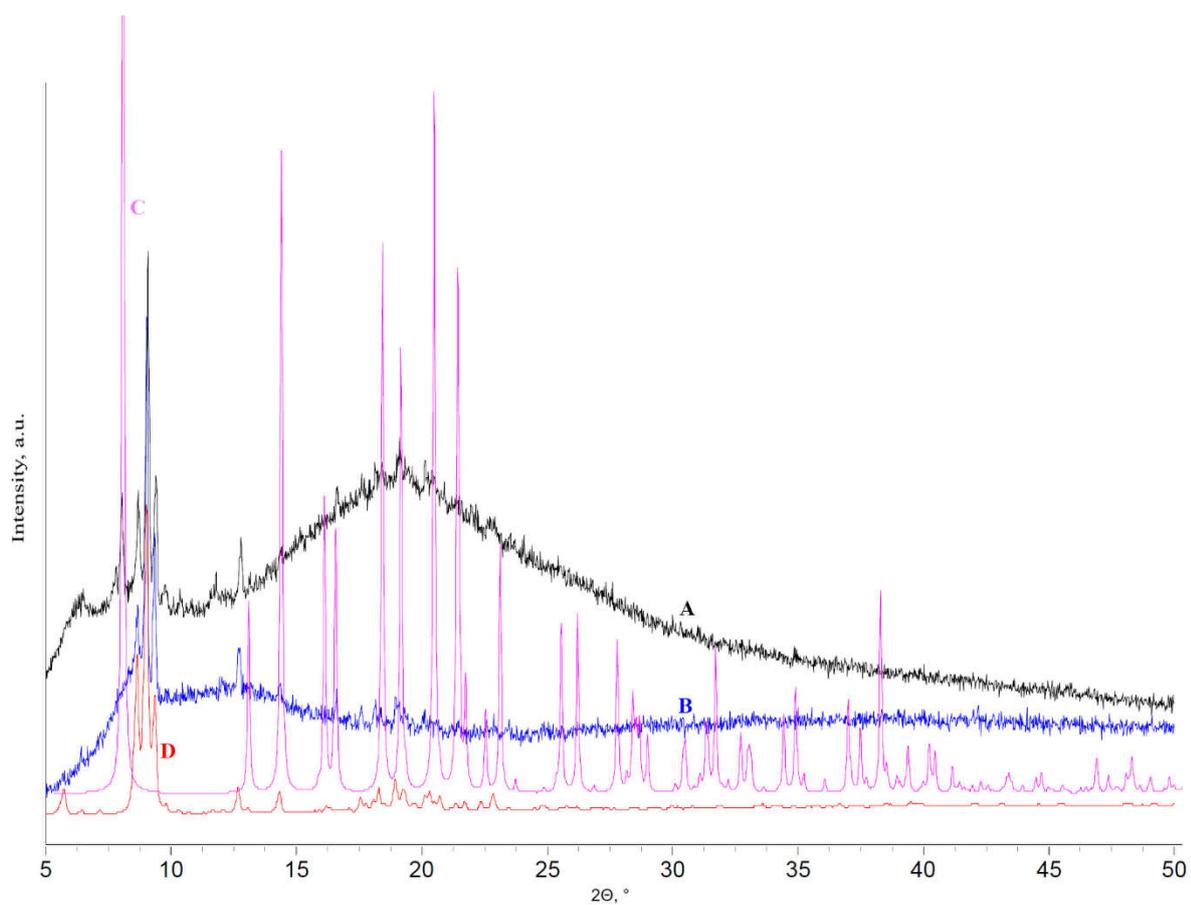


Figure S8. Comparison of experimental for sample 2 in time in air atmosphere (blue line (B) - after 40 min in air; black line (A) - after 2 months in air) and theoretical diffraction patterns for 2 (red line (C)) and 3 (pink line (D)).

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