Supporting Information for

Single-crystal FeFe(CN)₆ nanoparticles: A high capacity and

high rate cathode for Na-ion batteries

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1. Thermogravimetric analysis (TG) of the FeFe(CN)₆ sample:



Figure S1. Thermogravimetric curve of the $FeFe(CN)_6$ sample at a rate of 10 °C min⁻¹ in air.

Thermogravimetric analysis (Figure S1) reveals a large weight loss of 20% occurred in the temperature range of 50-150 °C, corresponding to four water molecules per $FeFe(CN)_6$ unit. The chemical composition of the sample can thus be expressed as $FeFe(CN)_6 \cdot 4H_2O$.

2. Infrared spectra (IR) of the FeFe(CN)₆ sample:



Figure S2. Infrared spectra of the FeFe(CN)₆ sample

From the IR spectrum of the FeFe(CN)₆ sample, a singlet peak was observed at 2090 cm⁻¹, corresponding to the characteristic of the C=N⁻ vibration in the lattice.

3. *Ex-situ* **XRD** patterns of the FeFe $(CN)_6$ electrodes charged or discharged at different depths



Figure S3. *Ex-situ* XRD patterns (right) of the $FeFe(CN)_6$ electrodes charged or discharged at different depths as labeled by Arabic characters on the charge-discharge curve (left).

Table 1. The lattice parameter of $FeFe(CN)_6$ electrodes during the Na⁺ insertion / extraction process

	At the points labeled at CV curves	Na content (x)	lattice parameter (a)
Na ⁺ insertion process	a	0	10.18
	b	0.5	10.21
	с	1	10.29
	d	1.52	10.41
Na ⁺ extraction process	е	0	10.19