Structural transition and superconductivity in hydrothermally synthesized Fe*X* (*X*=S,Se)

Supplementary

Table S1: Rietveld refinement of $FeSe_{hydro}$ in the triclinic low-temperature phase (10 K)

Space group		$P\bar{1}$ (Nr. 2)			
a (pm)		376.59(2)			
b (pm)		376.66(2)			
c (pm)		547.93(1)			
α (°)		90.024(4)			
β (°)		89.943(4)			
γ (°)		90.168(2)			
Volume (Å^3)		77.72(1)			
Atom	*Wyck.*	*x*	*y*	*z*	b_{eq}
Fe	2*i*	0.2328(7)	0.25*	0.00*	0.44(2)
Se	2*i*	0.7458(5)	0.25*	0.2663(1)	0.20(1)
R_{wp}		3.25			
R_{exp}		1.84			
X^2		1.76			
Fe - Fe (pm)		256.9(2), 257.7(2), 275.2(2), 276.0(2)			
Fe - Se (pm)		234.3(2), 238.35(3), 238.41(3), 242.2(2)			
Se-Fe-Se (°)		113.93(1), 114.14(1), 104.43(6), 104.38(1), 109.89(1), 110.14(1)			

*Coordinates fixed after refinements converged to $y = 0.25$ respectively $z = 0$ within the *esd's*.

Figure S1: Rietveld fit of $FeSe_{hydro}$ at 10 K. Insert: Splitting of the $(220)_{tetra}$ Bragg reflection into the doublet [$(220)_{triclinic}$, $(2\bar{2}0)_{triclinic}$].

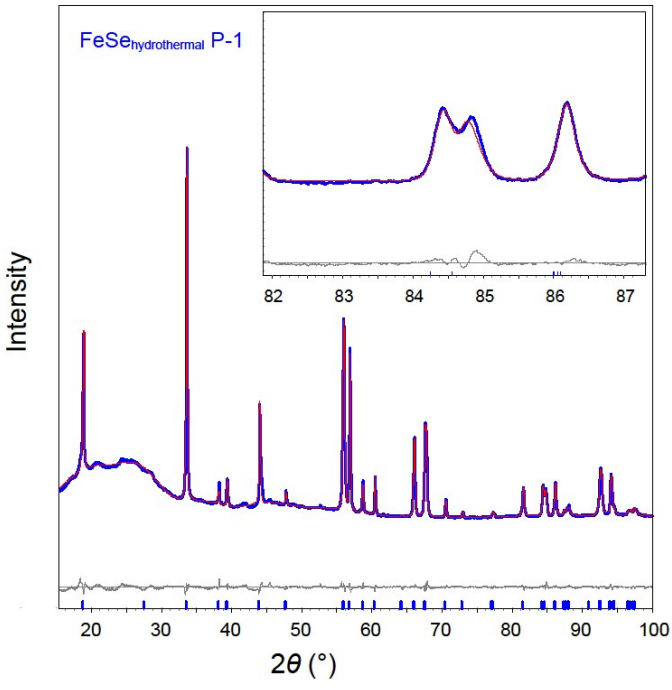
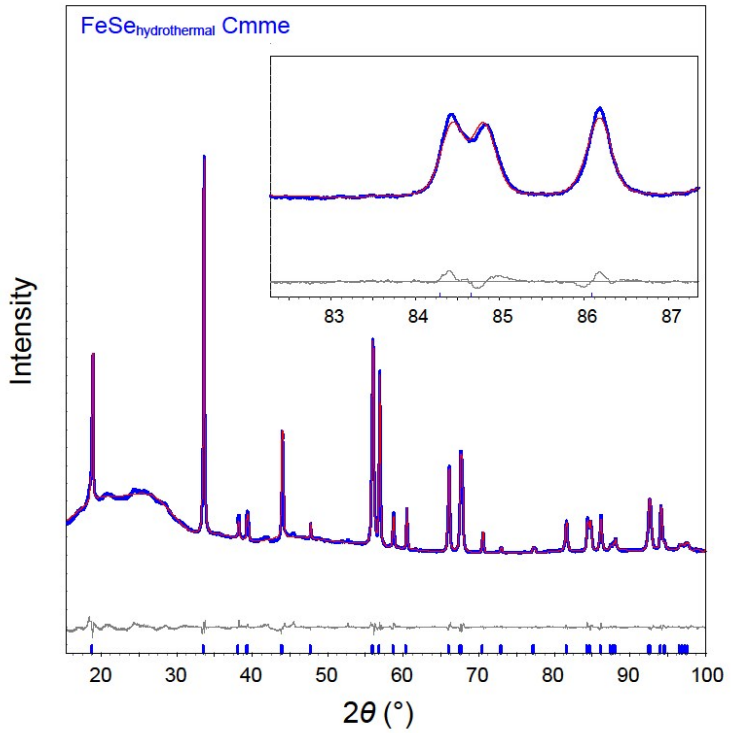


Figure S2: Rietveld-fits of $FeSe_{hydro}$ at 10 K: orthorhombic (*Cmme*) versus mixture of tetragonal (*P*4/*nmm*) and orthorhombic (*Cmme*) symmetry illustrated at the $(220)_{tetra}$ Bragg reflection splitted into the doublet [$(040)_{ortho}$, $(400)_{ortho}$].

a) Orthorhombic (*Cmme*)



b) Orthorhombic (*Cmme*) and tetragonal (*P*4/*nmm*) mixed

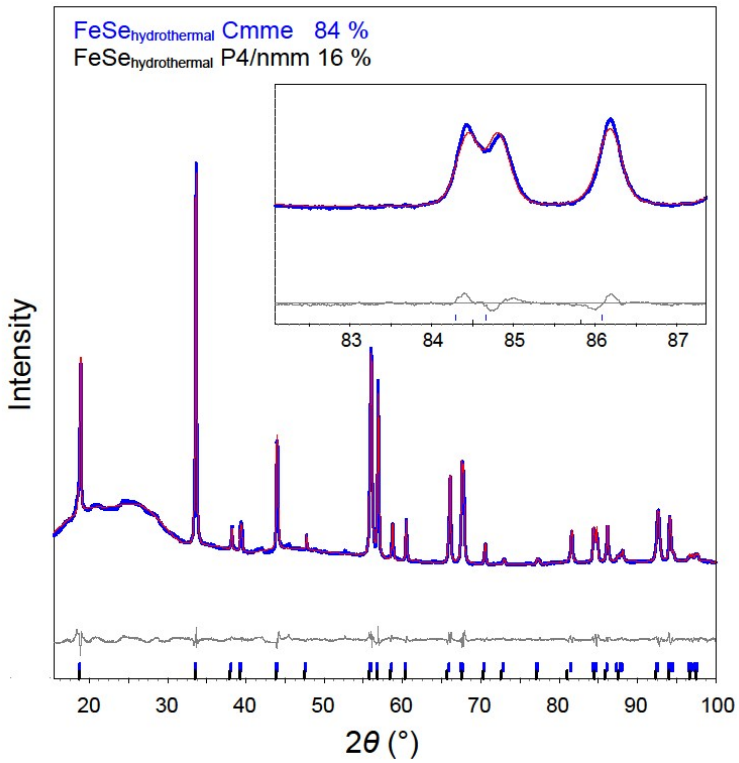


Figure S3: Temperature evolution of the unit cell volume in FeSe synthesized via hydrothermal (black) and conventional (blue) reaction method, respectively.

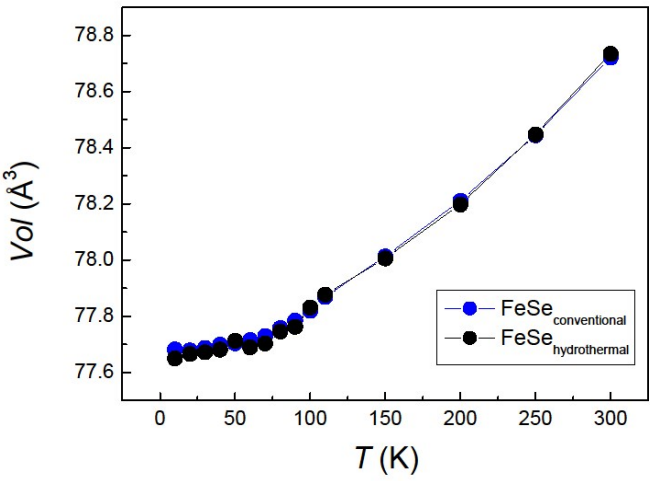


Table S2: Crystallographic data of FeS (single crystal)

Formula	FeS
Formula weight (g·mol^{-1})	87.9
Crystal System	Tetragonal
Space group	*P*4/*nmm* O2 (No. 129)
a, *c* (pm)	368.06(3), 502.83(7)
V (nm^3)	0.06812(1)
Z	2
d_{calc} (g·cm^3)	4.2859
μ (Mo-K$_\alpha$) (mm^{-1})	11.809
Crystal Size (μm^3)	20 × 20 × 2
Temperature (K)	293
Wavelength (pm)	λ = 71.073
θ range (deg.)	4.1 – 30.3
hkl range	–4 → +5; –5 → +4; –7 → +7
Tot., Uniq. Data, R_{int}	1248, 81, 0.108
N_{Refl}, N_{Par}	81, 6
$R1$, wR_2, S	0.0805, 0.0903, 1.99
$\Delta\rho_{min}$, $\Delta\rho_{max}$, /eÅ^{-3}	–3.79, 3.92

Atomic positions and equivalent displacement parameters (Å^2)

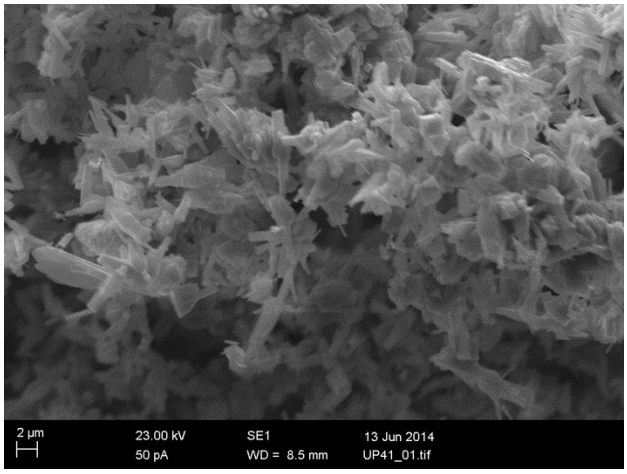
Atom	*Wyck*	*x*	*y*	*z*	U_{eq}
Fe	2*a*	0	0	0	0.0088(6)
S	2*c*	0	½	0.255(1)	0.0096(9)

Selected bond lengths (pm) and angles (deg)

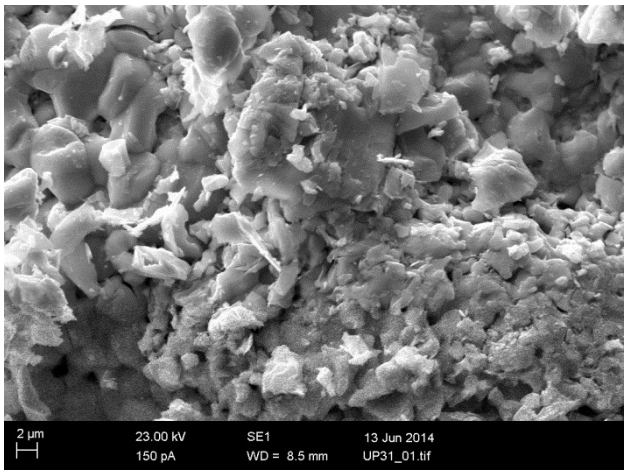
Fe-Fe	260.3(1)	S-Fe-S	110.3(1) ×2	109.1(1) ×4
Fe-S	224.3(1)			

Figure S4. SEM images of the samples showing morphology

a) hydrothermally prepared FeSe



b) conventionally prepared FeSe



c) hydrothermally prepared FeS