Supplementary Information

Systematic dimensional reduction of the layered FeSe structure by solvothermal synthesis

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Crystal structures

Tables 1S to 5S show the data for the crystal structure solutions of $Fe_{10}Se_{12}(en)_7$ and $Fe_3Se_4(en)_3$.

Table 1S Crystal data, data collection parameters and refinement parameters for $Fe_{10}Se_{12}(en)_7$ and $Fe_3Se_4(en)_3$.

	$Fe_{10}Se_{12}(en)_7$	$Fe_3Se_4(en)_3$
Crystal data		
Chemical formula	$Fe_{10}Se_{12}C_{14}H_{56}N_{14}$	$Fe_3Se_4C_6H_{24}N_6$
Formula mass	1926.7	663.7
Crystal system	Monoclinic	Triclinic
a [Å]	9.323 (2)	9.104 (4)
b [Å]	12.273 (2)	10.398 (4)
c [Å]	20.821 (4)	11.675 (5)
α [°]	90	109.880 (13)
β [°]	90	96.370 (15)
γ [°]	90	114.093 (14)
Unit cell volume [Å^3]	2382.4 (8)	907.8 (7)
Temperature [K]	120	293
Space group	$P2_1/n$	$P\bar{1}$
Z	2	2
Radiation type	Mo $K_{\alpha 1}$ radiation (λ = 0.71073 Å)	Mo $K_{\alpha 1}$ radiation (λ = 0.71073 Å)
Absorption coefficient μ [mm^{-1}]	12.15	10.38
Density D_{calc} [g·cm^{-3}]	2.686	2.428
$F(000)$	1812	632
θ range [°]	4.8–53.2	4.7–35.5
Data collection		
Diffractometer	Bruker CCD diffractometer	Bruker CCD diffractometer
Monochromator	graphite	graphite
Absorption correction	multi-scan *SADABS* 2014/5	multi-scan *SADABS* 2014/5
T (min; max)	0.696; 1.000	0.818; 1.000
No. of reflections measured	42237	37683
No. of independent reflections	4294	3280
No. of reflections with $I>3\sigma(I)$	3334	2424
R_{int}	0.058	0.100
Index range	$-12 \le h \le 12$, $-14 \le k \le 16$, $-27 \le l \le 27$	$-12 \le h \le 12$, $-13 \le k \le 13$, $-15 \le l \le 15$
θ range [°]	2.4-28.6	2.3-28.5
Refinement		
Refinement on	F^2	F^2
Data, restraints, parameters	4294, 0, 226	3280, 0, 172
H-atom treatment	parameters constrained	parameters constrained
Constraints	119	102
R_1 ($I>2\sigma(I)$)	0.033	0.038
$wR(F^2)$ ($I>2\sigma(I)$)	0.071	0.072
Goodness of fit on F^2	1.69	0.072
$\Delta\rho$ (max; min) [e·Å^{-3}]	0.98; -1.08	0.84; -0.84

Table 2S Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters [Å^2] for $Fe_{10}Se_{12}(en)_7$.

Atom	X	y	z	$U_{iso}*/U_{eq}$
Se1	0.77140 (6)	0.93036 (5)	0.12391 (3)	0.02419 (19)
Se2	0.85492 (6)	0.40006 (5)	0.03729 (3)	0.02412 (19)
Se3	0.11796 (6)	0.88151 (4)	0.03094 (3)	0.01944 (18)
Se4	0.76490 (6)	0.69621 (5)	0.99923 (3)	0.02219 (18)
Se5	0.99674 (7)	0.64847 (5)	0.14486 (3)	0.0288 (2)
Se6	0.93985 (7)	0.85096 (5)	0.86411 (3)	0.0260 (2)
Fe1	0.90711 (9)	0.79092 (6)	0.07937 (4)	0.0198 (3)
Fe2	0.94844 (9)	0.58040 (6)	0.04005 (4)	0.0199 (3)
Fe3	0.95131 (9)	0.02130 (6)	0.06705 (4)	0.0197 (3)
Fe4	0.99098 (9)	0.75666 (6)	0.95840 (4)	0.0193 (3)
Fe5	0.45243 (9)	0.42641 (7)	0.18158 (4)	0.0261 (3)
N1	0.4521 (6)	0.60599 (9)	0.19970 (17)	0.043 (2)
N2	0.4758 (3)	0.4916 (2)	0.08256 (9)	0.0344 (19)
N3	0.3853 (2)	0.3987 (3)	0.28151 (6)	0.0311 (18)
N4	0.21578 (13)	0.4177 (3)	0.16990 (10)	0.0306 (18)
N5	0.4870 (3)	0.24995 (11)	0.1676 (2)	0.051 (2)
N6	0.68602 (15)	0.4163 (2)	0.20335 (19)	0.041 (2)
N7	0.4303 (6)	0.06143 (16)	0.0768 (2)	0.069 (3)
C1	0.5133 (7)	0.6590 (5)	0.1416 (3)	0.043 (3)
C2	0.4422 (6)	0.6097 (5)	0.08454 (19)	0.039 (2)
C3	0.2348 (8)	0.3792 (9)	0.2812 (2)	0.150 (7)
C4	0.1513 (4)	0.4124 (6)	0.2330 (3)	0.061 (3)
C5	0.6462 (7)	0.2344 (4)	0.1631 (3)	0.045 (3)
C6	0.7244 (5)	0.3022 (6)	0.2091 (3)	0.050 (3)
C7	0.4977 (7)	0.0560 (5)	0.0156 (3)	0.049 (3)
H1n1	0.510766	0.629146	0.23993	0.0512*
H2n1	0.345824	0.629951	0.205316	0.0512*
H1n2	0.415562	0.454569	0.046631	0.0413*
H2n2	0.58438	0.481009	0.073014	0.0413*
H1n3	0.436756	0.33644	0.306372	0.0374*
H2n3	0.406264	0.472794	0.30385	0.0374*
H1n4	0.172051	0.482695	0.144506	0.0368*
H2n4	0.195726	0.345503	0.14536	0.0368*
H1n5	0.440502	0.235022	0.123091	0.0612*
H2n5	0.445073	0.196092	0.201339	0.0612*
H1n6	0.702192	0.455067	0.247175	0.049*
H2n6	0.74966	0.452857	0.168435	0.049*
H1n7	0.503517	0.089391	0.110717	0.0833*
H2n7	0.343562	0.114577	0.074623	0.0833*
H1c1	0.628282	0.643295	0.139485	0.0519*
H2c1	0.490702	0.746111	0.143017	0.0519*
H1c2	0.326484	0.620857	0.088239	0.0472*
H2c2	0.482091	0.648682	0.041016	0.0472*
H1c3	0.21437	0.293373	0.290961	0.1795*
H2c3	0.188844	0.405985	0.326726	0.1795*
H1c4	0.104481	0.491252	0.244894	0.0737*
H2c4	0.055269	0.361928	0.231049	0.0737*
H1c5	0.672258	0.148938	0.171287	0.0546*
H2c5	0.681859	0.253446	0.114514	0.0546*
H1c6	0.701903	0.274205	0.257757	0.06*
H2c6	0.839392	0.292721	0.201409	0.06*
H1c7	0.606155	0.088585	0.019117	0.0585*
H2c7	0.44655	0.113715	0.983101	0.0585*

Table 3S Atomic displacement parameters [Å^2] for Fe$_{10}$Se$_{12}$(*en*)$_7$.

Atom	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.0283 (3)	0.0210 (3)	0.0233 (3)	0.0008 (3)	0.0074 (3)	-0.0006 (3)
Se2	0.0257 (3)	0.0165 (3)	0.0301 (3)	-0.0007 (3)	0.0094 (3)	0.0005 (3)
Se3	0.0222 (3)	0.0180 (3)	0.0181 (3)	-0.0002 (3)	0.0005 (2)	0.0004 (2)
Se4	0.0236 (3)	0.0212 (3)	0.0218 (3)	-0.0019 (3)	0.0022 (3)	-0.0015 (3)
Se5	0.0463 (4)	0.0210 (3)	0.0190 (3)	0.0041 (3)	-0.0014 (3)	0.0014 (3)
Se6	0.0387 (4)	0.0232 (3)	0.0160 (3)	-0.0045 (3)	-0.0016 (3)	0.0008 (3)
Fe1	0.0259 (5)	0.0163 (4)	0.0172 (4)	0.0006 (4)	0.0032 (4)	0.0003 (3)
Fe2	0.0256 (5)	0.0160 (4)	0.0181 (4)	-0.0003 (4)	0.0048 (4)	0.0001 (3)
Fe3	0.0262 (5)	0.0156 (4)	0.0174 (4)	-0.0003 (4)	0.0014 (4)	0.0003 (3)
Fe4	0.0252 (5)	0.0164 (4)	0.0164 (4)	-0.0015 (4)	0.0025 (4)	0.0002 (3)
Fe5	0.0274 (5)	0.0284 (5)	0.0226 (5)	0.0026 (4)	-0.0021 (4)	-0.0033 (4)
N1	0.070 (4)	0.032 (3)	0.026 (3)	-0.010 (3)	0.010 (3)	0.000 (3)
N2	0.031 (3)	0.049 (4)	0.023 (3)	-0.004 (3)	-0.002 (2)	-0.007 (3)
N3	0.032 (3)	0.031 (3)	0.030 (3)	-0.006 (2)	-0.002 (2)	0.006 (2)
N4	0.029 (3)	0.038 (3)	0.025 (3)	0.002 (2)	-0.009 (2)	-0.001 (2)
N5	0.056 (4)	0.039 (4)	0.059 (4)	0.001 (3)	0.016 (3)	-0.013 (3)
N6	0.030 (3)	0.062 (4)	0.030 (3)	0.007 (3)	0.006 (3)	0.012 (3)
N7	0.043 (4)	0.083 (5)	0.082 (5)	0.003 (4)	-0.002 (4)	-0.031 (4)
C1	0.052 (5)	0.043 (4)	0.035 (4)	-0.006 (4)	0.013 (3)	0.006 (3)
C2	0.029 (4)	0.062 (5)	0.027 (4)	-0.011 (4)	-0.002 (3)	0.010 (3)
C3	0.041 (6)	0.37 (2)	0.035 (5)	-0.069 (9)	-0.008 (4)	0.051 (8)
C4	0.034 (4)	0.111 (8)	0.040 (4)	0.008 (5)	0.006 (4)	0.027 (5)
C5	0.044 (4)	0.037 (4)	0.056 (5)	0.010 (4)	0.024 (4)	-0.003 (4)
C6	0.052 (5)	0.055 (5)	0.042 (4)	0.013 (4)	0.004 (4)	0.000 (4)
C7	0.031 (4)	0.066 (5)	0.049 (5)	0.010 (4)	-0.007 (3)	-0.018 (4)

Table 4S Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters [Å^2] for Fe$_3$Se$_4$(*en*)$_3$.

Atom	X	y	z	U_{iso}*/U_{eq}
Se1	0.13526 (8)	0.74158 (7)	0.33777 (6)	0.0249 (3)
Se2	0.76829 (8)	0.35087 (7)	0.18216 (6)	0.0284 (3)
Se3	0.87130 (8)	0.59483 (7)	0.98113 (6)	0.0257 (3)
Se4	0.85343 (8)	0.57306 (8)	0.54767 (6)	0.0282 (3)
Fe1	0.98106 (10)	0.52543 (9)	0.12988 (8)	0.0198 (4)
Fe2	0.97611 (11)	0.52367 (9)	0.38322 (8)	0.0209 (4)
Fe3	0.66239 (11)	0.88136 (10)	0.24881 (8)	0.0267 (4)
N1	0.6610 (3)	0.10858 (14)	0.3256 (3)	0.030 (3)
N2	0.41219 (18)	0.8168 (2)	0.1413 (3)	0.033 (3)
N3	0.5870 (5)	0.8456 (2)	0.4131 (2)	0.030 (3)
N4	0.6037 (6)	0.63463 (13)	0.19656 (14)	0.049 (4)
N5	0.7703 (4)	0.9194 (2)	0.09406 (15)	0.040 (3)
N6	0.93694 (14)	0.9979 (4)	0.34486 (15)	0.038 (3)
C1	0.4897 (9)	0.0816 (5)	0.2853 (6)	0.038 (4)
C2	0.4062 (5)	0.9597 (7)	0.1500 (6)	0.035 (4)
C3	0.6015 (8)	0.7120 (8)	0.4181 (6)	0.043 (4)
C4	0.5258 (9)	0.5829 (5)	0.2884 (6)	0.043 (4)
C5	0.9439 (9)	0.0506 (8)	0.1552 (5)	0.044 (4)
C6	0.0332 (5)	0.0185 (8)	0.2511 (6)	0.043 (4)
H1n1	0.711662	0.176956	0.423205	0.0363*
H2n1	0.733733	0.165473	0.278053	0.0363*
H1n2	0.367751	0.736909	0.046421	0.04*
H2n2	0.337443	0.768982	0.192376	0.04*
H1n3	0.655531	0.941941	0.500158	0.036*
H2n3	0.461276	0.819483	0.394196	0.036*
H1n4	0.512961	0.580973	0.109347	0.0593*
H2n4	0.698706	0.604534	0.185081	0.0593*
H1n5	0.69875	0.948888	0.042567	0.0476*
H2n5	0.773238	0.82126	0.033008	0.0476*
H1n6	0.965726	0.933208	0.385647	0.0453*
H2n6	0.96936	0.106329	0.415612	0.0453*
H1c1	0.492512	0.189555	0.291245	0.0451*
H2c1	0.419493	0.041444	0.346932	0.0451*
H1c2	0.275584	0.933932	0.121519	0.0416*
H2c2	0.472478	0.00239	0.08776	0.0416*
H1c3	0.534642	0.674665	0.482229	0.0515*
H2c3	0.73376	0.745087	0.451608	0.0515*
H1c4	0.544052	0.486078	0.289816	0.0512*
H2c4	0.391059	0.542488	0.258467	0.0512*
H1c5	0.939579	0.158306	0.20412	0.053*
H2c5	0.009513	0.058085	0.083134	0.053*
H1c6	0.041162	0.912857	0.201805	0.0522*
H2c6	0.159459	0.116181	0.30098	0.0522*

Table 5S Atomic displacement parameters [Å^2] for Fe$_3$Se$_4$(*en*)$_3$.

Atom	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.0269 (4)	0.0177 (3)	0.0177 (3)	0.0017 (3)	0.0026 (3)	0.0062 (3)
Se2	0.0261 (4)	0.0251 (4)	0.0179 (3)	-0.0011 (3)	0.0010 (3)	0.0098 (3)
Se3	0.0318 (4)	0.0275 (4)	0.0211 (3)	0.0172 (3)	0.0071 (3)	0.0105 (3)
Se4	0.0342 (4)	0.0347 (4)	0.0235 (4)	0.0210 (3)	0.0113 (3)	0.0142 (3)
Fe1	0.0207 (5)	0.0179 (5)	0.0144 (4)	0.0049 (4)	0.0028 (4)	0.0060 (4)
Fe2	0.0232 (5)	0.0188 (5)	0.0154 (4)	0.0058 (4)	0.0034 (4)	0.0072 (4)
Fe3	0.0285 (5)	0.0260 (5)	0.0208 (5)	0.0095 (4)	0.0062 (4)	0.0094 (4)
N1	0.034 (3)	0.022 (3)	0.019 (3)	0.003 (3)	-0.001 (2)	0.006 (2)
N2	0.031 (3)	0.027 (3)	0.021 (3)	0.000 (3)	-0.002 (2)	0.007 (2)
N3	0.035 (3)	0.033 (3)	0.019 (3)	0.013 (3)	0.008 (2)	0.012 (2)
N4	0.070 (5)	0.043 (4)	0.049 (4)	0.034 (4)	0.028 (4)	0.021 (3)
N5	0.048 (4)	0.044 (4)	0.028 (3)	0.024 (3)	0.013 (3)	0.012 (3)
N6	0.032 (3)	0.040 (4)	0.041 (4)	0.016 (3)	0.008 (3)	0.019 (3)
C1	0.046 (5)	0.035 (4)	0.032 (4)	0.018 (4)	0.018 (3)	0.013 (3)
C2	0.025 (4)	0.043 (4)	0.036 (4)	0.013 (3)	0.010 (3)	0.021 (3)
C3	0.055 (5)	0.046 (5)	0.041 (4)	0.025 (4)	0.020 (4)	0.030 (4)
C4	0.054 (5)	0.030 (4)	0.045 (5)	0.021 (4)	0.013 (4)	0.017 (4)
C5	0.058 (5)	0.044 (5)	0.036 (4)	0.022 (4)	0.033 (4)	0.020 (4)
C6	0.034 (4)	0.038 (4)	0.052 (5)	0.016 (4)	0.013 (4)	0.013 (4)

Tables 6S and 7S show the data for the Rietveld refinement of the powder diffraction data of FeSe(*en*)$_{0.3}$. Positions of the C, N, and H atoms (*en* molecules) were taken from rigid body refinement, isotropic displacement factors were set to 3 and 5, respectively, and site occupation factors were set to one third in accordance with the chemical analysis.

Table 6S Crystal data, data collection parameters and refinement parameters for FeSe(*en*)$_{0.3}$.

	FeSe(en)$_{0.3}$
Crystal data	
Chemical formula	Fe$_{0.85}$SeC$_{0.6}$H$_{2.4}$N$_{0.6}$
Formula mass	144.46
Crystal system	Monoclinic
a [Å]	3.9037 (5)
b [Å]	21.528 (2)
c [Å]	3.8585 (6)
α [°]	90
β [°]	91.34 (2)
γ [°]	90
Unit cell volume [Å^3]	324.17 (8)
Space group	*C*2/*c*
Z	4
Radiation type	Mo $K_{\alpha 1}$ radiation (λ = 0.71073 Å)
Data collection	
Diffractometer	STOE STADI P diffractometer
Monochromator	Ge111
Refinement	
R_p	3.239
R_{wp}	4.294
Goodness of fit	1.219

Table 7S Fractional atomic coordinates, isotropic displacement parameters [Å^2] and site occupation factors for FeSe(en)$_{0.3}$.

Atom	X	y	z	B_{iso}	Occ.
Se1	0	0.1832 (3)	0.25	0.96 (19)	1
Fe1	0	0.2499 (6)	0.75	0.96 (19)	0.85
C1	0.19533	0.0035	0.0236	3	0.3333333
N1	0.3404	0.02628	0.69974	3	0.3333333
H1	0.25169	0.03627	0.23167	5	0.3333333
H2	0.30791	0.95879	0.0891	5	0.3333333
H5	0.47118	0.06683	0.76133	5	0.3333333
H6	0.52387	0.99422	0.62774	5	0.3333333

Chemical analysis

The chemical composition of the new compounds was verified by CHN elemental analysis (Table 8S) and energy-dispersive spectroscopy measurements EDS (Table 9S). The small deviation in C : H : N ratio compared to the formula of en $C_2H_8N_2$ is attributed to contamination with residues of acetone C_3H_6O from washing process which could not be fully removed.

Table 8S C : N : H ratio from elemental analysis normalized to N = 2.

	C	H	N
Fe$_3$Se$_4$(en)$_3$	2.1	7.9	2
Fe$_{10}$Se$_{12}$(en)$_7$	2.1	8.5	2
FeSe(en)$_{0.3}$	2.2	8.8	2

Table 9S Fe : Se ratio from EDS analysis normalized to Se.

	Fe	Se
Fe$_3$Se$_4$(en)$_3$	3.30(43)	4
Fe$_{10}$Se$_{12}$(en)$_7$	10.60(56)	12
FeSe(en)$_{0.3}$	0.95(7)	1

FeSe(en)$_{0.3}$ was further investigated by Thermogravimetric and ICP-OES analysis. The former showed that deintercalation of en starts near 200 °C and is completed at 230 °C under recovery of β-FeSe. The liquid section of the decomposed product was subsequently investigated by ^{1}H and ^{13}C NMR which showed pure en, thus no other molecular species had been intercalated. A molar ratio of FeSe : en = 3 : 1 was observed. ICP-OES analysis yielded a Fe : Se ratio of 0.84(5) : 1 and a FeSe : en ratio of 3 : 1. Due to the surface sensitivity of the EDS analysis we consider the ICP-OES results as most reliable and assume a total composition Fe$_{0.85(5)}$Se(en)$_{0.3}$.

Phase diagram

Table 10S shows the composition of the products from solvothermal synthesis between 160 °C and 220 °C and 0 % and 50 % glycerol content calculated from powder diffraction data by Rietveld refinement.

Temperature [°C]	% glycerol	Fe$_3$Se$_4$(*en*)$_3$	Fe$_3$Se$_4$(*en*)$_2$	Fe$_{10}$Se$_{12}$(*en*)$_7$	FeSe(*en*)$_{0.3}$	β-FeSe	FeSe$_2$	Se	Fe
					wt.-%				
220	0	-	100	-	-	-	-	-	-
220	20	-	-	26	74	-	-	-	-
220	50	-	-	-	99	-	-	1	-
210	0	-	100	-	-	-	-	-	-
210	20	-	-	100	-	-	-	-	-
210	50	-	-	-	89	9	-	2	-
200	0	-	100	-	-	-	-	-	-
200	5	-	57	43	-	-	-	-	-
200	20	-	-	100	-	-	-	-	-
200	30	-	-	100	-	-	-	-	-
200	50	-	-	-	100	-	-	-	-
190	0	14	57	29	-	-	-	-	-
190	20	-	-	97	-	-	-	3	-
190	50	-	-	13	68	-	17	3	-
180	0	60*	6*	34*	-	-	-	-	-
180	20	-	-	100	-	-	-	-	-
180	50	-	-	8	83		9	-	-
170	0	99*	-	-	-	-	-	-	1*
170	20	-	-	(100)	-	-	-	-	-
170	50	-	-	-	94	-	-	6	
160	0	(83)*	-	-	-	-	-	(6)*	(11)*
160	20	-	-	(94)	-	-	-	(6)	-
160	50	-	-	-	74	21	-	4	-

Samples marked with * contain a so far unknown phase.

Properties

Table 11S shows the lattice parameters of Fe$_3$Se$_4$(*en*)$_3$, Fe$_3$Se$_4$(*en*)$_2$, Fe$_{10}$Se$_{12}$(*en*)$_7$ and FeSe(*en*)$_{0.3}$ between 10 K and 290 K from temperature dependent powder diffraction data determined by Rietveld refinement.

Table 11S Lattice parameters of $Fe_3Se_4(en)_3$, $Fe_3Se_4(en)_2$, $Fe_{10}Se_{12}(en)_7$, and $FeSe(en)_{0.3}$ determined by Rietveld refinement from low temperature powder diffraction data (Co-K$_{\alpha 1}$ radiation) between 10 K and 290 K.

	$Fe_3Se_4(en)_3$					
T [K]	a [Å]	b [Å]	c [Å]	α [°]	β [°]	γ [°]
290	9.1246(5)	10.3659(8)	11.6972(13)	109.841(8)	96.443(8)	114.025(5)
270	9.1050(5)	10.3611(7)	11.6974(12)	109.908(7)	96.412(7)	113.962(5)
250	9.0772(5)	10.3621(7)	11.7027(12)	110.001(7)	96.344(7)	113.927(5)
230	9.0728(6)	10.3571(8)	11.6927(11)	110.038(7)	96.302(7)	113.897(5)
210	9.0665(6)	10.3455(9)	11.6885(12)	110.073(8)	96.280(8)	113.813(6)
190	9.0488(6)	10.3413(8)	11.6873(12)	110.118(7)	96.265(7)	113.766(5)
170	9.0292(5)	10.3424(7)	11.6867(10)	110.178(6)	96.230(7)	113.735(5)
150	9.0162(5)	10.3368(7)	11.6861(10)	110.246(7)	96.201(7)	113.681(4)
130	9.0011(5)	10.3287(7)	11.6875(11)	110.304(7)	96.182(8)	113.624(5)
110	8.9875(5)	10.3243(7)	11.6875(10)	110.359(7)	96.176(7)	113.577(5)
90	8.9750(5)	10.3187(7)	11.6888(10)	110.405(7)	96.172(7)	113.534(5)
70	8.9637(5)	10.3153(7)	11.6875(10)	110.463(6)	96.145(7)	113.492(5)
50	8.9532(5)	10.3102(7)	11.6881(11)	110.515(7)	96.130(7)	113.450(5)
30	8.9438(5)	10.3087(7)	11.6932(11)	110.581(7)	96.105(7)	113.412(5)
10	8.9429(5)	10.3126(7)	11.6953(10)	110.599(7)	96.085(7)	113.401(5)
	$Fe_3Se_4(en)_2$					
T [K]	a [Å]	b [Å]	c [Å]	α [°]	β [°]	γ [°]
290	17.2758(4)	7.9768(2)	11.6789(4)	90	120.938(2)	90
270	17.2742(4)	7.9708(3)	11.6743(5)	90	120.959(3)	90
250	17.2695(4)	7.9652(2)	11.6670(5)	90	120.964(2)	90
230	17.2656(4)	7.9586(2)	11.6590(4)	90	120.976(2)	90
210	17.2653(3)	7.9530(2)	11.6549(4)	90	121.001(2)	90
190	17.2606(3)	7.9493(2)	11.6463(3)	90	121.001(2)	90
170	17.2540(3)	7.9417(2)	11.6398(4)	90	121.011(2)	90
150	17.2451(4)	7.9361(2)	11.6348(4)	90	121.034(2)	90
130	17.2361(4)	7.9292(2)	11.6282(4)	90	121.030(2)	90
110	17.2290(4)	7.9245(2)	11.6240(4)	90	121.046(2)	90
90	17.2231(4)	7.9212(2)	11.6212(4)	90	121.053(2)	90
70	17.2167(4)	7.9174(2)	11.6185(4)	90	121.057(2)	90
50	17.2084(4)	7.9136(2)	11.6127(4)	90	121.058(2)	90
30	17.2060(4)	7.9124(2)	11.6106(4)	90	121.053(2)	90
10	17.2084(4)	7.9131(2)	11.6121(4)	90	121.058(2)	90
	$Fe_{10}Se_{12}(en)_7$					
T [K]	a [Å]	b [Å]	c [Å]	α [°]	β [°]	γ [°]
290	9.4176(6)	12.3271(20)	20.9802(20)	90	90	90
270	9.4076(6)	12.3207(23)	20.9684(22)	90	90	90
250	9.3968(6)	12.3125(22)	20.9508(21)	90	90	90
230	9.3847(6)	12.3077(22)	20.9356(22)	90	90	90
210	9.3739(6)	12.3055(22)	20.9176(22)	90	90	90
190	9.3661(6)	12.2986(22)	20.9071(22)	90	90	90
170	9.3597(6)	12.2950(23)	20.8892(22)	90	90	90
150	9.3509(7)	12.2902(24)	20.8730(23)	90	90	90
130	9.3456(7)	12.2978(32)	20.8580(24)	90	90	90
110	9.3407(7)	12.3057(38)	20.8506(24)	90	90	90
90	9.3327(7)	12.3124(43)	20.8430(24)	90	90	90
70	9.3248(7)	12.3128(44)	20.8346(24)	90	90	90
50	9.3186(7)	12.3064(43)	20.8291(24)	90	90	90
30	9.3083(7)	12.3062(41)	20.8231(24)	90	90	90
10	9.3131(7)	12.3074(42)	20.8311(23)	90	90	90

T [K]	a [Å]	b [Å]	c [Å]	α [°]	β [°]	γ [°]
	FeSe(*en*)$_{0.3}$					
290	3.9153(3)	21.5280(17)	3.8603(3)	90	91.354(12)	90
270	3.9133(3)	21.4964(17)	3.8630(3)	90	91.644(13)	90
250	3.9123(3)	21.4632(17)	3.8643(3)	90	91.776(12)	90
230	3.9108(3)	21.4396(16)	3.8652(3)	90	91.795(12)	90
210	3.9095(3)	21.4139(16)	3.8639(3)	90	91.826(12)	90
190	3.9082(3)	21.3886(16)	3.8643(3)	90	91.821(12)	90
170	3.9063(3)	21.3764(16)	3.8634(3)	90	91.808(11)	90
150	3.9044(3)	21.3566(17)	3.8625(3)	90	91.816(12)	90
130	3.9035(3)	21.3374(16)	3.8615(3)	90	91.808(11)	90
110	3.9018(3)	21.3244(16)	3.8597(3)	90	91.820(11)	90
90	3.9002(3)	21.3128(16)	3.8595(3)	90	91.820(11)	90
70	3.8991(3)	21.2995(16)	3.8576(3)	90	91.800(10)	90
50	3.8986(3)	21.2889(16)	3.8578(3)	90	91.807(10)	90
30	3.8982(3)	21.2833(16)	3.8564(3)	90	91.806(10)	90
10	3.8985(3)	21.2727(16)	3.8569(3)	90	91.823(11)	90

Figure 1S shows the isothermal magnetization curves of $Fe_3Se_4(en)_3$, $Fe_{10}Se_{12}(en)_7$ and FeSe(en)$_{0.3}$ at 1.8 K and between 1 and 5 T with μ in μ_B per formula unit.
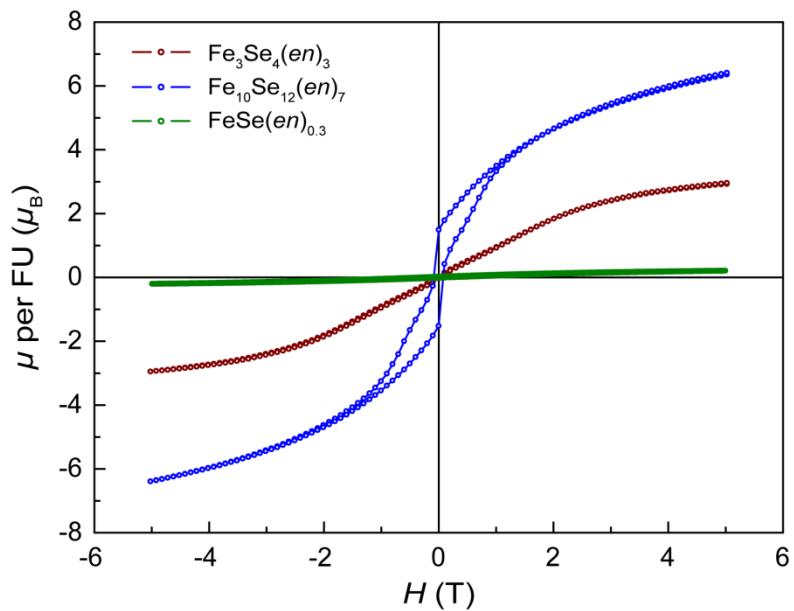

Figure 1S Isothermal magnetization curves of $Fe_3Se_4(en)_3$, $Fe_{10}Se_{12}(en)_7$ and FeSe(en)$_{0.3}$ at 1.8 K.