Table S1. Reported Conditions for the Solution-Phase Synthesis of Group 3-5 Chalcogenide Nanomaterials[a]

Material	Methodology	Metal Precursor(s)	Chalcogenide Precursor	Temp.[b]	Reaction Time[c]	Solvent/Ligands[d]	Stoichiometry (M:(M'):E)[e]	Total Solvent[f]	Scale (mmol)[g]	Yield[h]	Morphology/Size	Ref.
$NaYS_2$	Continuous supply of H_2S gas at high temp.	Na(acac), $Y(acac)_3$	H_2S	280 °C	1 h	HDA, ODE (1:1 mol:mol)	2:1:16	20 mmol	0.5		Hexagonal plates, 50-100 nm	1
TiS_2	Heat-up	$TiCl_4$	Elemental S	215 °C	12 h	OlAm	1:2	9 mL	0.91		Single-layer nanodiscs (50 nm)	2
							1:4				Single-layer nanodiscs (34 nm)	
					1 h		1:2				Single-layer nanodiscs (18 nm)	
TiS_2	Hot-injection of titanium	$TiCl_4$	Elemental S	300 °C	10 min	ODE	1:6	20 mL	1.0		Flower-like clusters (non-colloidal)	3
	Hot-injection of titanium followed by heat-up			Injection at 150 °C then heat-up to 300 °C							Clusters of nanoflakes (non-colloidal)	
TiS_2	Hot-injection of titanium followed by heat-up	$TiCl_4$	Elemental S	Injection at 250 °C then heat-up to 300 °C	90 min	OlAm, OlAc (7:1 vol:vol)	1:6	16 mL	1.0		Inorganic fullerene nanoparticles (range of sizes/shapes)	4
TiS_2	Heat-up			300 °C							Hollow inorganic fullerene nanospheres (250-400 nm)	
TiS_2	Heat-up	$TiCl_4$	Elemental S	300 °C	3 h	OlAm	1:12.5	5 mL	0.4		Multilayer nanosheets (~500 nm X ~5 nm)	5
						DDA					Nanorods	

Material	Methodology	Metal Precursor(s)	Chalcogenide Precursor	Temp.[b]	Reaction Time[c]	Solvent/Ligands[d]	Stoichiometry (M:(M'):E)[e]	Total Solvent[f]	Scale (mmol)[g]	Yield[h]	Morphology/Size	Ref.
						TOPO					“random” shapes and sizes of nanoparticles	
TiS_2	Hot-injection of sulfur precursor	$TiCl_4$	CS_2	300 °C	15 min	OlAm	1:3.3	11.2 mmol	2.0		Nanodiscs (~100 nm)	6
									1.5		Nanodiscs (~150 nm)	
									3.6		Nanodiscs (~40 nm)	
TiS_2	Heat-up	$TiCl_4$	DDT	230 °C	11 h	OlAm	1:19	18.7 mmol	0.26		Single-layer nanosheets (300 nm – 1 µm)	7
$TiSe_2$	Heat-up	$TiCl_4$	Se	300 °C	30 min	OlAm	1:2	6.0 mmol	0.5		Hexagonal plates, 250 nm X 30 nm	6
ZrS_2	Hot injection of sulfur precursor	$ZrCl_4$	CS_2	300 °C	1 h	OlAm	1:3.3	18.7 mmol	1.5	~100 mg	Nanodiscs (20 nm)	8
					3 h						Nanodiscs (35 nm)	
					6 h						Nanodiscs (60 nm)	
ZrS_2	Heat-up	$ZrCl_4$	DDT	245 °C	10 h	OlAm	1:16.7	18.7 mmol	0.3		Single-layer nanosheets (200-500 nm)	7
$ZrSe_3$	Heat-up	$ZrCl_4$	Se	300 °C	1 h	OlAm	1:2	14.9 mmol	0.73		Irregular nanoplatelets (20 nm)	6
HfS_2	Hot injection of sulfur precursor	$HfCl_4$	CS_2	300 °C	12 h	OlAm	1:6.9	11.2 mmol	0.73		Nanodiscs (20 nm)	6
HfS_2	Heat-up	$HfCl_4$	DDT	245 °C	10 h	OlAm	1:14.8	18.7 mmol	0.77		Single-layer nanosheets (~500 nm)	7
$HfSe_3$	Heat-up	$HfCl_4$	Se	300 °C	12 h	OlAm	1:2	11.2 mmol	0.73		Irregular nanoplatelets (90 nm)	6

Material	Methodology	Metal Precursor(s)	Chalcogenide Precursor	Temp.[b]	Reaction Time[c]	Solvent/Ligands[d]	Stoichiometry (M:(M'):E)[e]	Total Solvent[f]	Scale (mmol)[g]	Yield[h]	Morphology/Size	Ref.
$BaTiS_3$	Hot-injection of sulfur precursor	$Ba\{N(SiMe_3)_2\}_2(THF)_2$, $Ti(NMe_2)_4$	N,N'-diethyl-thiourea	360 °C	30 min	OlAm	1:1:30	3.7 mL	0.1		Nanorods (60-100 nm X ~6 nm)	9
	Heat-up				2 h						Roughly spherical nanoparticles (~10-20 nm)	
$BaTiS_3$	Heat-up	$Ba(S_2CNR_2)_2$, $Ti(S_2CN^iPr_2)_4$	--[i]	350 °C	30 min	OlAm	2:1:--[i]	3.7 mL	0.05		Nanorods	10
$BaZrS_3$	Heat-up	$Ba\{N(SiMe_3)_2\}_2(THF)_2$, $Zr(NMe_2)_4$	N,N'-diethyl-thiourea	365 °C	1 h	OlAm	1:2:60	1.2 mL (0.08 M in Ba^{2+})	0.1	48%	Platelets (~20 nm)	11
$BaZrS_3$	Heat-up	$Ba(S_2CNBu_2)_2$, $Zr(S_2CNEt_2)_4$	--[i]	330 °C	0.5 – 18 h	OlAm	1:1:--[i]	1 mL	0.244		Multicrystalline platelet-like particles	12
VS_2	Hot-injection of sulfur precursor	VCl_4	CS_2	330 °C	6 h	OlAm	1:6.9	18.7 mmol	0.73		Nanoplatelets (18 nm)	6
VS_2	Heat-up	$V(acac)_3$	S	330 °C	6 h	OlAm	1:2.4	25 mL	0.5		Nanoplatelets (~10-30 nm)[k]	13
							1:9.6				Nanoplatelets (~50-60 nm)[k]	
VSe_2	Heat-up	VCl_4	Se	300 °C	1 h	OlAm	1:2	11.2 mmol	0.73		Hexagonal nanoplatelets (150 nm)	6
VSe_2	Heat-up	$V(O)(acac)_2$	Se	330 °C	1.5 h	OlAm	1:2	10 mL	0.3		Monolayer nanosheets (80 nm)	14
								(not given)	40	1.36 g		
VSe_2	Hot-injection of selenium precursor followed by heat-up	$V(O)(acac)_2$	Se	Injection at 140 °C then heating to 250 °C	2 h	OlAm, DDT (18:1 vol:vol)	1:2	19 mL	0.5		Nanosheets	15
VSe_2	Heat-up	$V(O)(acac)_2$	Se	300 °C	4 h	OlAm, TDA (1:1 vol:vol)	1:2.7	100 mL	1.5	(1.16 g, 91%)[m]	Aggregated nanosheets	16

Material	Methodology	Metal Precursor(s)	Chalcogenide Precursor	Temp.[b]	Reaction Time[c]	Solvent/Ligands[d]	Stoichiometry (M:(M'):E)[e]	Total Solvent[f]	Scale (mmol)[g]	Yield[h]	Morphology/Size	Ref.
VSe_2	Slow hot-injection of both precursors into hot solvent	VCl_3	$(PhCH_2)_2Se_2$	280 °C	30 min	OlAm, ODE (15:5 vol:vol)	2:1	20 mL	0.5		Clusters of nanoflakes[i]	17
NbS_2	Hot-injection of sulfur precursor	$NbCl_5$	CS_2	300 °C	3 h	OlAm	1:6.9	11.2 mmol	0.73		Irregular nanoparticles (100 nm)	6
NbS_2	Hot-injection of sulfur precursor	$NbCl_5$	CS_2	300 °C	3 h	OlAm	1:60	6 g	1		Clusters of nanosheets[k]	18
NbS_2	Hot-injection of sulfur precursor	$NbCl_5$	CS_2	300 °C	3 h	OM	1:35	20 mL	0.23		Stacked multilayer nanosheets	19
					2 h	OlAc, OlAm (0.3:1 mol:mol)	1:35				Hexagonal nanoplatelets, (106 X 9.5 nm)	
					2 h	OlAc, OlAm (0.3:1 mol:mol)	1:70				Nanohexagons (55 nm)	
					2 h	OlAc, OlAm (0.3:1 mol:mol)	1:140				Hexagonal nanorods (55 X 127 nm)	
NbS_2	Hot-injection of sulfur precursor	$NbCl_5$	CS_2	280 °C	2 h	OlAm	1:10	8 mL	1.0		Ultrathin nanosheets (~2 µm)	20
				300 °C			1:10				Ultrathin nanosheets (~3 µm)	
				280 °C			1:60				Stacked nanodisks (~300 nm)	
				300 °C			1:60				Stacked nanodisks (~500 nm)	
$NbSe_2$	Heat-up	$NbCl_5$	Se	280 °C	4 h	OlAm	1:2	20 mL	1		Depends on cooling rate:	21

Material	Methodology	Metal Precursor(s)	Chalcogenide Precursor	Temp.^{b}	Reaction Time^c	Solvent/Ligands^d	Stoichiometry (M:(M'):E)^e	Total Solvent^f	Scale (mmol)^g	Yield^h	Morphology/Size	Ref.
				250 ^{o}C		DDA					nanoplates (slow cooling) or nanowires (fast cooling)^k	
$NbSe_2$	Heat-up	$NbCl_5$	Se	300 ^{o}C	2 h	OM	1:2	11.2 mmol	0.73		Irregular nanoparticles (45 nm)	6
$NbSe_2$	Heat-up	$NbCl_5$	Se, SeO_2, or selenourea	320 ^{o}C	2 h	OM	1:4	0.125 M concentration	(not given)		Nanosheets; oxide impurities present when Se or SeO_2 used as precursor	22
$NbSe_2$	Hot-injection of metal precursor	$NbCl_5$	Se	300 ^{o}C	1 h	OlAm, ODE (16:5 vol:vol)	1:2	21 mL	0.5		Nanosheets	23
Nb_2Se_9	Heat-up	NbF_5	Se, SeO_2, or selenourea	320 ^{o}C	2 h	OlAm	1:4	0.125 M concentration	-- (not given)		Nanorods	22
Nb_2Se_9	Heat-up	$NbCl_5$	Se	300 ^{o}C	30 min	OlAm, ODE, TOP (35:35:4 vol:vol)	1:5.6	74 mL	2.0		Flower-like nanoclusters^k	24
Nb_2Se_9	Heat-up	$NbCl_5$	Se	280 ^{o}C	2 h	ODE	2:9	20 mL	0.6	57%	Nanorods (20-100 nm)	25
						OlAm			0.6	40%	Flower-like nanosheet aggregates	
						ODT			7.5	64%	Irregular agglomerated nanoparticles	
						ODE, OlAm (16:4 vol:vol)			0.6	34%	Microwires (7 µm)	
TaS_2	Hot-injection of sulfur precursor	$TaCl_5$	CS_2	300 ^{o}C	1.5 h	OlAm	1:6.9	11.2 mmol	0.73		Irregular nanoparticles (120 nm)	6

Material	Methodology	Metal Precursor(s)	Chalcogenide Precursor	Temp.[b]	Reaction Time[c]	Solvent/Ligands[d]	Stoichiometry (M:(M'):E)[e]	Total Solvent[f]	Scale (mmol)[g]	Yield[h]	Morphology/Size	Ref.
TaS_2	Hot-injection of sulfur precursor	$TaCl_5$	CS_2	300 °C	2 h	OlAm, ODE, DDT (12:6:2 vol:vol)	1:8	20 mL	1.0		Nanoflakes (~150 nm)	26
TaS_2	Hot-injection	$TaCl_5$	CS_2	300 °C	1.5 h	OlAm	1:8	4 mL	1.17		Aggregated nanosheets; morphology/aggregation depends on degassing/drying method	27
$TaSe_2$	Heat-up	$TaCl_5$	Se	300 °C	1 h	OlAm	1:2	11.2 mmol	0.73		Irregular nanoparticles (150 nm)	6
$TaSe_2$	Heat-up	$TaCl_5$	Se	305 °C	1 h	OlAm, ODE (6:9 vol:vol)	1:2	15 mL	1.0		Folded nanosheets	26
Cu_3VS_4	Hot-injection of sulfur precursor	$V(O)(acac)_2$, $Cu(acac)_2$	S	230 °C	30 min	OlAm	2.5:6:8	40 mL	2.0		Irregularly-shaped nanoparticles (~10 nm)[k]	28
Cu_3VS_4	Hot-injection of sulfur precursor	$V(O)(acac)_2$, CuI	DDT	280 °C	30 min	OlAm, ODE, TOP (1:7:0.45 vol:vol)	1.33:1:10	8.45 mL	0.5		Nanocubes (18 nm)	29
				250 °C							Nanocubes (9 nm)	
Cu_3VSe_4	Hot-injection of copper precursor	VSe_2 (pre-formed *in situ*), $CuCl_2$	--[i]	250 °C	1 h	OlAm, DDT (23:1 vol:vol)	1:1.6:2	24 mL	0.5		Nanosheets	15
Cu_3NbS_4	Hot-injection of copper precursor	$CuCl_2$, NbS_2 (pre-formed nanosheets)	CS_2[n]	300 °C	2 h	OlAm, DDT (25:1 vol:vol)	3:1:33	26 mL	0.5		Irregularly-shaped nanoparticles	23
Cu_3NbSe_4	Hot-injection of copper precursor	$CuCl_2$, $NbSe_2$ (pre-formed nanosheets)	--[i]	300 °C	1 h	OlAm, ODE (22:5 vol:vol)	3:1:4	27 mL	0.5		Irregularly-shaped nanoparticles	23
Cu_3NbSe_4	Hot-injection of selenium precursor	CuCl, $NbCl_5$	Ph_2Se_2	280 °C	30 min	OlAm	3:1:4	8 mL	0.1		Nanocubes (12 nm)	30
					60 min						Nanocubes (15 nm)	
Cu_3TaS_4		CuCl, $TaCl_5$	CS_2	300 °C	30 min	OlAm	3:1:9.2	14.8 mL	1.5		Nanocubes (20 nm) and	31

Material	Methodology	Metal Precursor(s)	Chalcogenide Precursor	Temp.[b]	Reaction Time[c]	Solvent/Ligands[d]	Stoichiometry (M:(M'):E)[e]	Total Solvent[f]	Scale (mmol)[g]	Yield[h]	Morphology/Size	Ref.
	Hot-injection of sulfur precursor										irregular particles	
							1:1:7.7		1.0		Nanocubes (15 nm)	
Cu_3TaS_4	Hot-injection of sulfur precursor	$CuCl_2\cdot 2H_2O$, $TaCl_5$	CS_2	300 °C	1 h	OlAm, ODE, DDT (17:6:2 vol:vol)	2:1:5.5	25 mL	1.0		Nanocubes (15 nm)	26
Cu_3TaS_4	Hot-injection of copper precursor	$CuCl_2\cdot 2H_2O$, TaS_2 (pre-formed nanosheets)	--[i]	300 °C	1 h	OlAm, ODE, DDT (17:6:2 vol:vol)	1.55:1:8.3	25 mL	0.5		Nanocubes (20 nm)	26
Cu_3TaSe_4	Hot-injection of metal precurosrs	$CuCl_2\cdot 2H_2O$, $TaCl_5$	Se	300 °C	1 h	OlAm, ODE (17:8 vol:vol)	1:1:2	25 mL	0.3		Ill-defined core-shell particles (~20 nm)	26
Cu_3TaSe_4	Hot-injection of copper precursor	$CuCl_2\cdot 2H_2O$, $TaSe_2$ (pre-formed nanosheets)	--[i]	305 °C	30 min	OlAm, ODE (14:6 vol: vol)	1.45:1:2	20 mL	0.5		Irregularly-sized nanocubes	26

[a] Table gives an overview of reaction time, temperature, stoichiometry, and concentrations. Other aspects of the procedure (such as injection rate, heating rate, degassing procedure, work-up, etc) are not included here but may be important to the reaction outcome; the original reference should be consulted for additional details. Some sources described the optimization/study of a range of different conditions; a subset of representative examples are included in the table and are grouped together.

Abbreviations: OlAm = oleylamine; ODE = octadecene; OlAc = oleic acid; DDT = dodecanethiol; ODT = octadecanethiol; HDA = hexadecylamine; DDA = dodecylamine; TDA = tetradecylamine; TOP = trioctylphosphine; TOPO = trioctylphosphine oxide.

[b] Temperature corresponds to the final reaction temperature after any prior drying/degassing and heat-up steps, unless otherwise specified.

[c] Listed time corresponds to the time the reaction is held at the final reaction temperature, not including the time for any prior drying/degassing and heat-up steps.

[d] Gives the final solvent/ligand composition after all precursor solutions have been combined; ratio is given as either vol/vol or mol/mol in accordance with what is given in the original reference.

[e] For ternary materials, metals are given in the same order as listed in the “metal precursors” column. For sulfide reactions including DDT as a co-solvent in addition to a more reactive sulfide source such as CS_2, DDT is not included in the sulfur stoichiometry although it could potentially act as a source of sulfur in the reaction. For reactions using elemental sulfur, stoichiometry is based on moles of S (not S_8). For cascade reactions starting from pre-formed but not isolated nanosheets, given stoichiometry includes the amount of precursors used in the first step to form the nanosheets.

[f] Total volume after all precursor solutions have been combined, given in either mL or mmol according to how it was reported in the original reference.

[g] Scale is given as theoretical yield of the product material.

[h] Most references do not report yield; where given in terms of mass or percent yield, it is provided here as reported.

[i] Procedure used a “single source precursor” where the chalcogenide source is also one of the metal precursors.

[k] Solid-state annealing step was carried out after solution-phase synthesis.

[m] A yield of 1.1593 g (91%) was given for a “gram-scale” version of the synthesis, but further details for the scaled-up reaction were not explicitly provided.

[n] Additional CS_2 was added after the preparation of the pre-formed nanosheets.

References:

1 Y. Ding, J. Gu, T. Zhang, A.-X. Yin, L. Yang, Y.-W. Zhang and C.-H. Yan, *J. Am. Chem. Soc.*, 2012, **134**, 3255–3264.
2 K. H. Park, J. Choi, H. J. Kim, D.-H. Oh, J. R. Ahn and S. U. Son, *Small*, 2008, **4**, 945–950.
3 S. Prabakar, C. W. Bumby and R. D. Tilley, *Chem. Mater.*, 2009, **21**, 1725–1730.
4 S. Prabakar, S. Collins, B. Northover and R. D. Tilley, *Chem. Commun.*, 2010, **47**, 439–441.
5 V. V. Plashnitsa, F. Vietmeyer, N. Petchsang, P. Tongying, T. H. Kosel and M. Kuno, *J. Phys. Chem. Lett.*, 2012, **3**, 1554–1558.
6 S. Jeong, D. Yoo, J. Jang, M. Kim and J. Cheon, *J. Am. Chem. Soc.*, 2012, **134**, 18233–18236.
7 D. Yoo, M. Kim, S. Jeong, J. Han and J. Cheon, *J. Am. Chem. Soc.*, 2014, **136**, 14670–14673.
8 J. Jang, S. Jeong, J. Seo, M.-C. Kim, E. Sim, Y. Oh, S. Nam, B. Park and J. Cheon, *J. Am. Chem. Soc.*, 2011, **133**, 7636–7639.
9 D. Zilevu and S. E. Creutz, *Chem. Mater.*, 2021, **33**, 5137–5146.
10 N. E. Ingram, B. J. Jordan, B. Donnadieu and S. E. Creutz, *Dalton Trans.*, 2021, **50**, 15978–15982.
11 D. Zilevu, O. O. Parks and S. E. Creutz, *Chem. Commun.*, 2022, **58**, 10512–10515.
12 R. Yang, A. D. Jess, C. Fai and C. J. Hages, *J. Am. Chem. Soc.*, 2022, **144**, 15928–15931.
13 Z. Guo, L. Yang, W. Wang, L. Cao and B. Dong, *J. Mater. Chem. A*, 2018, **6**, 14681–14688.
14 W. Zhao, B. Dong, Z. Guo, G. Su, R. Gao, W. Wang and L. Cao, *Chem. Commun.*, 2016, **52**, 9228–9231.
15 M. Liu, C.-Y. Lai, M. Zhang and D. R. Radu, *Sci. Rep.*, 2020, **10**, 21679.
16 W. Feng, M. Cheng, R. Du, Y. Wang, P. Wang, H. Li, L. Song, X. Wen, J. Yang, X. Li, J. He and J. Shi, *Adv. Mater. Interfaces*, 2022, **9**, 2200060.
17 J. Zhang, J. Li, H. Huang, W. Chen, Y. Cui, Y. Li, W. Mao, X. Zhu and X. Li, *Small*, 2022, **18**, 2204557.
18 J. Zhang, C. Du, Z. Dai, W. Chen, Y. Zheng, B. Li, Y. Zong, X. Wang, J. Zhu and Q. Yan, *ACS Nano*, 2017, **11**, 10599–10607.
19 A. Mansouri and N. Semagina, *ACS Appl. Nano Mater.*, 2018, **1**, 4408–4412.
20 W. Li, X. Wei, H. Dong, Y. Ou, S. Xiao, Y. Yang, P. Xiao and Y. Zhang, *Front. Chem.*, 2020, **8**, 189.
21 P. Sekar, E. C. Greyson, J. E. Barton and T. W. Odom, *J. Am. Chem. Soc.*, 2005, **127**, 2054–2055.
22 T. Kolokoto, V. Mashindi, R. Kadzutu-Sithole, L. F. E. Machogo-Phao, Z. B. Ndala, N. P. Shumbula, S. S. Nkabinde, G. N. Ngubeni, S. S. Gqoba, K. P. Mubiayi and N. Moloto, *RSC Adv.*, 2021, **11**, 31159–31173.
23 C.-Y. Chang, R. Prado-Rivera, M. Liu, C.-Y. Lai and D. R. Radu, *ACS Nanosci. Au*, 2022, **2**, 440–447.
24 X. Wu, B. Wu, H. Wang, Q. Zhuang, Z. Xiong, H. Yi, P. Xu, G. Shi, Y. Guo and B. Wang, *Energy Fuels*, 2021, **35**, 11563–11571.
25 T. Y. Kim, C. Woo, K. H. Choi, X. Dong, J. Jeon, J. Ahn, X. Zhang, J. Kang, H.-S. Oh, H. K. Yu and J.-Y. Choi, *Nanoscale*, 2022, **14**, 17365–17371.
26 M. Liu, C.-Y. Lai, C.-Y. Chang and D. R. Radu, *Crystals*, 2021, **11**, 51.
27 W. Shen, L. Qiao, J. Ding and Y. Sui, *J. Alloys Compd.*, 2023, **935**, 167877.
28 C.-C. Chen, K. H. Stone, C.-Y. Lai, K. D. Dobson and D. Radu, *Mater. Lett.*, 2018, **211**, 179–182.
29 V. Mantella, S. Ninova, S. Saris, A. Loiudice, U. Aschauer and R. Buonsanti, *Chem. Mater.*, 2019, **31**, 532–540.
30 Y. Zhao, M. Liu, W. Zhang, X. Sun, W. Wang, W. Zhang, M. Tang, W. Ren, M. Sun, W. Feng and W. Wang, *Dalton Trans.*, 2022, **51**, 16937–16944.
31 A. Haque, S. Ershadrad, T. D. Chonamada, D. Saha, B. Sanyal and P. K. Santra, *J. Mater. Chem. A*, 2022, **10**, 19925–19934.