

## **Electronic Supporting Information**

### **A review on the recently developed promising infrared nonlinear optical materials**

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## 1. The summary of synthesis methods for IR NLO materials

**High temperature solid-state reaction method in the sealed system:** The concrete steps of synthesis are as follows: Starting reactants were weighed and ground under oxygen and water-free atmosphere, then the mixture was put into a tidy silica tube that was washed with deionized water and dried in high-temperature to remove the inside impurities, and the tube was flame-sealed under  $10^{-3}$  Pa (When synthesizing the alkali or alkali earth metal chalcogenides, the reaction was performed in a polished graphite crucible, located inside the quartz ampoule for avoiding the corrosion of quartz ampoule). The tube was heated to the appropriate temperature at an average rate of  $20\text{ }^{\circ}\text{C}/\text{h}$ , and kept at that temperature for several days, followed by slowly cooled to room temperature at an average rate of  $3\sim 5\text{ }^{\circ}\text{C}/\text{h}$ . Finally, the product was carefully washed with N,N-dimethylformamide (DMF) or distilled water to remove other byproducts and then dried with ethanol.

The reaction temperature is a very important factor for material exploration, for example, the lower temperature or fast cooling always leads to poor crystallization, and higher temperatures or rapid heating always arouse the explosion of the quartz tube. From Table S1, it is found that the alkali earth or transition metal chalcogenides show the relatively higher reaction temperature, and substitution of alkali metal elements can decrease the temperature because the low melting points of alkali metals can serve to solubilize the starting reagents at lower temperatures, such as the  $\text{T}_2\text{BaMQ}_4$  and  $\text{BHgMQ}_4$  ( $\text{T} = \text{Ag, Cu}$ ;  $\text{B} = \text{Sr, Ba}$ ;  $\text{M} = \text{Si, Ge, Sn}$ ;  $\text{Q} = \text{S, Se}$ ) were synthesized at high temperatures ( $\sim 900\text{ }^{\circ}\text{C}$ ), the  $\text{A}_2\text{BaMQ}_4$  and  $\text{Li}_2\text{HgMQ}_4$  ( $\text{A} = \text{Li, Na}$ ;  $\text{M} = \text{Si, Ge, Sn}$ ;  $\text{Q} = \text{S, Se}$ ) were synthesized at lower temperatures about  $600\sim 800\text{ }^{\circ}\text{C}$ . Moreover, the flux can also decrease the reaction temperature very effectively, for example,  $\text{Sn}_2\text{Ga}_2\text{S}_5$  were obtained from heating the mixture of starting materials at  $950\text{ }^{\circ}\text{C}$ , and it was also obtained by a facile mid-temperature ( $450\text{ }^{\circ}\text{C}$ ) fluxing method using  $\text{SnCl}_2$  as the flux.  $\text{CuBa}_4\text{Ga}_5\text{Q}_{12}$  were prepared by using stoichiometric amounts of starting reactants with a flux KBr at a relatively lower temperature of  $750\text{ }^{\circ}\text{C}$ , which temperature is much lower than those of isostructural compounds  $\text{LiBa}_4\text{Ga}_5\text{Q}_{12}$  ( $1000\text{ }^{\circ}\text{C}$ ). Besides, the investigations of chalcoborates were mainly focused on exploring the synthetic utility of ionic salts AX ( $\text{A} = \text{Li, Na, K, Rb, Cs, Ca, Sr, Ba, Pb}$  etc.;  $\text{X} = \text{F, Cl, Br, I}$ ) acting as the flux (Table S1). The chalcoborates also show excellent IR NLO

properties, and only a few chalcoborates have been reported. Compared to other materials, synthesis of chalcoborate is relatively difficult because the high chemical reactivity of boron sulphide can attack on the silica tube and react with the silica tube, according to the reaction  $2B_2S_3 + 3SiO_2 = 2B_2O_3 + 3SiS_2$ . Therefore, the carbon-coated silica tube and carbon crucibles were necessary to apply during the synthesis of chalcoborate.

**High temperature solid-state reaction in the open system:** The high temperature solid-state reaction method in an open system is widely used for preparations of oxides, rather than in sealed ampoules. The recently reported POC ( $Pb_{17}O_8Cl_{18}$ ,  $Pb_{13}O_6Cl_4Br_{10}$ ,  $Pb_{13}O_6Cl_7Br_7$ ,  $Pb_{13}O_6Cl_9Br_5$ , and  $Pb_{18}O_8Cl_{15}I_5$ ) system was synthesized by starting reactants weighed at the corresponding molar ratio, and placed in an alumina crucible. The prepared reactant was gradually heated to the appropriate temperature (about 430 °C) and dwelled at that temperature for several hours for homogenization. Then, the temperature was cooled at a rate of 1 °C/h until the spontaneously nucleated crystals were observed. Besides, there are other oxide materials including  $Li_3VO_4$ ,  $A_2LiVO_4$  (A = Rb, Cs),  $Li_2ZrTeO_6$ ,  $Rb_4Li_2TiOGe_4O_{12}$ ,  $Li_2K_4TiOGe_4O_{12}$  in the IR region (Table S3). They were obtained by grinding the carbonate with other oxides together and then packed into a porcelain crucible. The temperature was raised slowly to the appropriate temperature at a rate of 2 °C/min in order to avoid ejection of starting materials from the crucible owing to vigorous evolution of  $CO_2$ , and the samples were ground again for facilitating the completion of the reactions.

**Hydrothermal or solvothermal syntheses:** Hydrothermal or solvothermal synthetic methods are widely applied in the synthesis of metal halide, oxide, and iodates, etc. The most of iodates listed in Table S3 were obtained by mixing the iodic acid, carbonates, nitrates, and other oxides with the water in an autoclave equipped with a Teflon liner (Table S3), which was heated at about 230 °C for several hours, followed by slowly cooling to room temperature at a rate of 1.5 °C/h. The reaction product was washed with cold water and dried in the air. Besides, several iodates were synthesized by solvent evaporation, such as  $NaI_3O_8$  was obtained by evaporation at 60 °C of a solution containing  $NaIO_3$  in nitric acid. The iodates  $Mn(IO_3)_2$  and  $Zn(IO_3)_2$  synthesized by metallic salts are dissolved in nitric acid; then 2 equivalents of lithium iodate, dissolved in nitric acid and added dropwise to the former solution. These mixtures are slowly evaporated at 60 °C.

## **2. The summary of measurement and theoretical calculations for IR NLO materials**

**NLO property measurement:** The SHG coefficients are one of the most important parameters for practical IR NLO materials. At present, two methods are widely applied to evaluate NLO properties of new materials. For bulk single crystal, the SHG matching and Maker fringes methods are employed to evaluate more accurate SHG coefficients effectively. However, in the absence of large single crystals, the typical powder SHG measurement method based on the modified Kurtz and Perry technique serves as a preliminary estimation of the SHG efficiencies for new polycrystalline samples. As early as 1968, Kurtz and Perry first proposed the typical measurement methods on powdery samples, and Kurtz later modified this method by detecting the second-harmonic laser with a photomultiplier and displaying it with an oscilloscope. With the development of technology, the powder SHG measurement method was significantly improved through modification of utilizing laser with tunable wavelengths and using a spectrometer and a CCD array detector. Finally, phase-matching and non-phase-matching behaviors are determined by comparing SHG intensities of various materials in different particle sizes. For powder SHG measurements of IR-NLO crystals, the crystals were ground and sieved into five different particle sizes, and the samples were placed on a glass microscope cover slide. Then, the commonly used incident lasers of 1.9, 2.05, and 2.1  $\mu\text{m}$  are employed to record SHG signals. The commercial material AGS is generally used as a reference sample.

**LIDT measurement:** Ideally, LIDT measurements should be evaluated on bulk single crystals by the most effective standard method, but the large crystal is often difficult to be available, and it may take a long time to obtain good quality crystals. Thus, the judgment of LIDTs on polycrystalline samples was carried out by the single-pulse method introduced by Guo et al. In this method, target samples and references were first sieved with the same particle size ranges, then packed into a plastic holder with a similar thickness (for example, 1 mm) between two glass microscope cover slides. After that, target samples and references were irradiated to high-power laser radiation successively. Then, the constantly apparent changes with increasing laser energies were observed by an optical microscope after a single pulse radiation every time. When the

obvious damage spot was discovered, the LIDT value of the samples can be estimated by calculating the laser powder and the damage spot area.

**Infrared spectrum measurement:** Mid-IR transparent regions including two important atmospheric transparent windows of 3–5  $\mu\text{m}$  and 8–12  $\mu\text{m}$  have significant applications in many military and civil activities. The evaluation of IR absorption edges is very essential for new material exploration. Indeed, high-quality single crystals are a prerequisite for IR transmission spectra test, but the measurements are proceeded based on the pressed pellets of the powder sample mixed with KBr because in short of a bulk single crystal. During the test, the powder samples and KBr were mixed in the ratio of about 1:100, dried, and ground into fine powder, and then pressed into a transparent sheet on the tablet machine. The sheet was loaded in the sample chamber, and then the IR spectrum was collected.

**Theoretical calculation:** With the aid of theoretical calculations, NLO properties could be obtained to help analyze the NLO properties of new materials and better understand the relationship between the structures and properties. At the earliest, the theoretical NLO properties were calculated by limited computation of bond parameter methods, and Levine's model based on the fundamental building units consisting of  $sp^3$ -hybrid tetrahedra is successfully used in A-B type compounds. Later, Chen et al. developed a theoretical model named "anionic group theory", then an ab initio method with the CASTEP package was performed to calculate the NLO properties. Besides, Song et al. calculated the frequency-dependent SHG coefficients by employing first-principles theoretical calculations based on the plane wave (FLAPW) method with the screened exchange local density approximation (sx-LDA) as well as the Hedin-Lundqvist form of the exchange-correlation potential (LDA). In this way, the calculations, including dipole moments, electronic band structures, SHG coefficients, and birefringence, have achieved significant success in structure–property relations and structural exploration, which will dramatically contribute to the future development of NLO and other material fields.

**Table S1.** The summary of recently developed chalcogenide IR NLO materials. high temperature solid state in sealed system (HTS), temperature (T), stoichiometric ratios (SR), second haremomic generation response (SHG), band gap ( $E_g$ ).

Compounds Q (Q = S, Se)	Method	T (°C)	flux	Molar ratio	raw materials	SHG (*AGS)	$E_g$ (eV)	Ref.
AgGaQ <sub>2</sub>	HTS	1050		SR	Ag:Ga:Q	1/3	2.73/1.83	1
LiGaQ <sub>2</sub>	HTS	850		SR	Li:Ga:Q	0.5/1	4.15/3.34	2
SnGa <sub>4</sub> Q <sub>7</sub>	HTS	850		SR	Sn:Ga:Q	1.3/3.8	3.10/2.55	3
BaAl <sub>4</sub> Q <sub>7</sub>	HTS	900		SR	BaS:Al <sub>2</sub> Q <sub>3</sub>	0.5/0.5	3.95/3.40	4,5
BaGa <sub>4</sub> S <sub>7</sub>	HTS	900		SR	BaS:Ga:S	0.4	3.54	6
BaGa <sub>4</sub> Se <sub>7</sub>	HTS	800		SR	Ba:Ga <sub>2</sub> Se <sub>3</sub> :Se	1.8	2.64	7
PbGa <sub>4</sub> S <sub>7</sub>	HTS	1000	Bi <sub>2</sub> S <sub>3</sub>	SR	PbQ:Ga <sub>2</sub> S <sub>3</sub>	1.2	3.08	8
ZnGa <sub>2</sub> S <sub>4</sub>	HTS	1200		SR	Zn:Ga:S	0.9	3.6	9
Cu <sub>2</sub> SiS <sub>3</sub>	HTS	800		SR	Cu <sub>2</sub> S:Si:S	1	2.31	10
Cu <sub>2</sub> SnS <sub>3</sub>	HTS	1100		SR	Cu:Sn:S	4.5	1.30	11
Na <sub>2</sub> Ge <sub>2</sub> Se <sub>5</sub>	HTS	550		SR	Na <sub>2</sub> Se:Ge:Se	3.6	2.38	12
Ag <sub>2</sub> GeS <sub>3</sub>	HTS	800		SR	Ag:Ge:S	3	1.98	13,14
Ba <sub>8</sub> Sn <sub>4</sub> S <sub>15</sub>	HTS	800		SR	BaS:SnS:SnS <sub>2</sub>	10	2.31	15
Ba <sub>6</sub> Sn <sub>6</sub> Se <sub>13</sub>	HTS	800		SR	BaSe:Sn:Se	0.3	1.52	16
A <sub>2</sub> SnS <sub>5</sub> (A = Ba, Sr)	HTS	830	K <sub>2</sub> S	SR	AS:Sn:S	1.1/0.3	2.38	17
Sn <sub>2</sub> Ga <sub>2</sub> S <sub>5</sub>	HTS	450	SnCl <sub>2</sub>	SR	Sn:Ga:S	2.5	2.14	18,19
AgGaGeS <sub>4</sub>	HTS	1080		SR	Ag:Ga:Ge:S	0.7	2.78	20
AHgSnS <sub>4</sub> (A = Sr Ba)	HTS	1000		SR	AS:HgS:SnS <sub>2</sub>	1.9/2.8	2.72/2.77	21
AHgSnSe <sub>4</sub> (A = Sr Ba)	HTS	900		SR	ASe:HgSe:SnSe <sub>2</sub>	4.9/5.1	2.07/1.98	21
AHgGeSe <sub>4</sub> (A = Sr Ba)	HTS	900		SR	ASe:HgSe:GeSe <sub>2</sub>	4.8/4.7	2.50	22
BaZnMSe <sub>4</sub> (M = Si:Ge)	HTS	900		SR	BaSe:ZnSe:M:Se	0.3/1	2.71/2.50	23
BaCdGeS <sub>4</sub> , BaMnSnS <sub>4</sub>	HTS	875		SR	Ba:Cd/Mn:Ge/Sn:S	0.3/1.2	2.58/1.9	24
EuCdGeQ <sub>4</sub>	HTS	1000		SR	EuQ:CdQ:GeQ <sub>2</sub>	2.6/3.8	2.50/2.25	25
EuHgGeSe <sub>4</sub> , EuHgSnS <sub>4</sub>	HTS	900		SR	Eu:HgQ:GeSe <sub>2</sub> :SnS <sub>2</sub>	1.97/2.14	3.1/1.77	26
SrCdGeQ <sub>4</sub>	HTS	950		SR	Sr:Cd:Ge:Q	2/5	2.60/1.90	27
ACdSnQ <sub>4</sub> (A=Sr,Ba)	HTS	850		SR	A:Cd:Sn:Q	1.3/1.5/1.6/0.7	2.05/1.54/1.79/2.30	28-30
SrZnSnS <sub>4</sub>	HTS	800		SR	SrS:Zn:Sn:S	0.5	2.83	31
ACd <sub>4</sub> Ga <sub>5</sub> S <sub>12</sub> (A = K, Rb, Cs)	HTS	1000	ACl	SR	ACl:Cd:Ga:S	12.2/11.1/9.8	2.98/3.02/3.09	32
ACd <sub>4</sub> Ga <sub>5</sub> Se <sub>12</sub> (A = K, Rb, Cs)	HTS	1000	ACl	3:4:5:12	ACl:Cd:Ga:Se	26.8/19.3/16.6	2.16/2.19/2.21	33
AZn <sub>4</sub> Ga <sub>5</sub> S <sub>12</sub> (A = K, Rb, Cs)	HTS	1100	ABr	4:5:12:15	Zn:Ga:S:ABr	1.4/1.3/1.2	3.65	34
AZn <sub>4</sub> In <sub>5</sub> Se <sub>12</sub> (A =Rb, Cs)	HTS	950	ABr	4:4:5:12	ABr:Zn:In:Se	3.9/3.5	2.09/2.11	35
ACd <sub>4</sub> In <sub>5</sub> Se <sub>12</sub> (A = Rb, Cs)	HTS	1000	ACl	3:4:5:12	ACl:Cd:In:Se	39.2/35.1	1.57/1.62	33
KHg <sub>4</sub> Ga <sub>5</sub> Se <sub>12</sub>	HTS	950	K <sub>2</sub> Se	3:8:5	K <sub>2</sub> Se:HgSe:Ga <sub>2</sub> Se <sub>3</sub>	20	1.61	36
LiBa <sub>4</sub> Ga <sub>5</sub> Q <sub>12</sub>	HTS	1000		3:4:5:12	Li:Ba:Ga:Q	1.5	3.43/2.55	37
CuBa <sub>4</sub> Ga <sub>5</sub> Q <sub>12</sub>	HTS	750	KBr	SR	Cu:Ba:Ga:Q	2.7/1.1	2.82/1.45	38
AgCd <sub>2</sub> GaS <sub>4</sub>	HTS	1147		SR	Ag:Cd:Ga:S	2	2.32	39
BaAg <sub>2</sub> MS <sub>4</sub> (M=Ge,Sn)	HTS	900	BaCl	SR	Ag:Ba:M:S	1.7/0.4	2.02/1.77	40
Ag <sub>2</sub> BaMSe <sub>4</sub> (M=Si,Ge,Sn)	HTS	1000		SR	Ag:Ba:M:Se	0.1~0.2	1.83/1.57/1.42	41
BaCu <sub>2</sub> MS <sub>4</sub> (M=Ge,Sn)	HTS	1050		1:2:1:3.5	BaS:Cu:M:S	0.3/1.6	2.47/1.96	42

BaCu <sub>2</sub> MSe <sub>4</sub> (M=Si, Ge, Sn)	HTS	1000		SR	Ba:Cu:M:S	0.3	2.62/1.88/1.72	42
Na <sub>2</sub> BaMQ <sub>4</sub> (M=Ge, Sn)	HTS	600		SR	Na:Ba:M:Q	0.5/0.9/1.3	3.27/2.46/2.25	43
Na <sub>2</sub> BaGeS <sub>4</sub>	HTS	700		1:6:2:8	Na:Ba:Ge:S	0.3	3.70	43
Li <sub>2</sub> BaMQ <sub>4</sub> (M=Ge, Sn)	HTS	800		SR	Li:Ba:M:Q	0.5/0.7/1.5/1.3	3.66/3.07/2.40/2.18	44
A <sub>2</sub> SrGeS <sub>4</sub> (A=Li, Na)	HTS	850		SR	A:SrS:Ge:S	0.5	3.75/3.8	45
A <sub>2</sub> SrSnS <sub>4</sub> (A=Li, Na)	HTS	750		SR	A:SrS:Sn:S	0.8	3.10/3.80	45
Li <sub>2</sub> ZnSiS <sub>4</sub>	HTS	900		SR	Li:Zn:Si:S	1.1	3.9	46
$\alpha/\beta$ -Li <sub>2</sub> ZnGeS <sub>4</sub>	HTS	700/900		SR	Li <sub>2</sub> S/Li:Zn/ZnS:Ge:S	3/0.7	4.09/3.49	47,48
Li <sub>2</sub> HgMS <sub>4</sub> (M = Si, Ge, Sn)	HTS	700		SR	Li:HgS:M:S	0.8/3/4	2.68/2.46/2.32	49
Li <sub>2</sub> CdSiS <sub>4</sub>	HTS	850		2:0.55:1:3.5	Li:CdS:Si:S	0.7	3.76	50
Li <sub>2</sub> MGeS <sub>4</sub> (M=Cd, Mn)	HTS	750	Li <sub>2</sub> S	SR	Li <sub>2</sub> S:M:Ge:S	2/0.5	3.15/3.07	51,52
Li <sub>4</sub> HgGe <sub>2</sub> S <sub>7</sub>	HTS	700		2:1:1:3	Li:HgS:Ge:S	1.5	2.75	53
Li <sub>4</sub> HgSn <sub>2</sub> Se <sub>7</sub>	HTS	600		SR	Li:HgSe:Sn:Se	3.6	2.10	54
Na <sub>2</sub> ZnSnS <sub>4</sub>	HTS	600		SR	Na <sub>2</sub> S <sub>2</sub> :Zn:Sn:S	0.9	3.10	55
K <sub>2</sub> MnGe <sub>3</sub> S <sub>8</sub>	HTS	800		1:01:03	K <sub>2</sub> S:MnS:GeS <sub>2</sub>	0.6	2.95	56
Na <sub>2</sub> Hg <sub>3</sub> M <sub>2</sub> S <sub>8</sub> (M=Si, Ge, Sn)	HTS	700		SR	Na:HgS:M:S	1.3/2.2/2.8	2.86/2.68/2.45	57
Na <sub>2</sub> ZnGe <sub>2</sub> S <sub>6</sub>	HTS	700		SR	Na:Zn:Ge:S	0.9	3.25	58
Na <sub>2</sub> CdGe <sub>2</sub> Q <sub>6</sub>	HTS	850		SR	Na:CdQ:Ge:Q	0.8/2	3.21/2.37	59
Ba <sub>2</sub> Ga <sub>8</sub> MS <sub>16</sub> (M=Si, Ge)	HTS	980		SR	Ba:Ga <sub>2</sub> S <sub>3</sub> :M:S	0.9	3.4/3.0	60
BaGa <sub>2</sub> GeSe <sub>6</sub>	HTS	850		SR	Ba:Ga:Ge:Se	3.5	2.22	61
BaGa <sub>2</sub> SiSe <sub>6</sub>	HTS	700		SR	BaSe: Ga <sub>2</sub> Se <sub>3</sub> :Si:Se	2.5	2.88	61
BaGa <sub>2</sub> MS <sub>6</sub> (M=Si, Ge)	HTS	900		SR	BaS:Ga <sub>2</sub> S <sub>3</sub> :MS <sub>2</sub>	1	3.75/3.23	61
SnGa <sub>2</sub> GeS <sub>6</sub>	HTS	1000		SR	SnS:Ga <sub>2</sub> S <sub>3</sub> :GeS <sub>2</sub>	0.25	2.04	62
PbGa <sub>2</sub> MSe <sub>4</sub> (M=Si, Ge)	HTS	800		SR	PbSe:Ga <sub>2</sub> Se <sub>3</sub> :M:Se	-1.5	2.17/1.96	63
RbMSn <sub>2</sub> Se <sub>6</sub> (M=Ga, In)	HTS	700		2:1.5:1.625:6	RbCl:M:Sn:Se	4.2/4.8	1.92/1.80	64
CsMSn <sub>2</sub> Se <sub>6</sub> (M=Ga, In)	HTS	800		1.3:1.775:6:2	M:Sn:Se:CsCl	3.5/4.0	1.87/1.78	65
Sr <sub>5</sub> ZnGa <sub>6</sub> S <sub>15</sub>	HTS	1050		SR	Sr:Zn:Ga:S	1.2	3.15	66
Ba <sub>6</sub> Zn <sub>7</sub> Ga <sub>2</sub> S <sub>16</sub>	HTS	850		SR	BaS:ZnS:Ga <sub>2</sub> S <sub>3</sub>	0.5	3.50	67
Ba <sub>10</sub> Zn <sub>7</sub> In <sub>6</sub> S <sub>26</sub>	HTS	950	KBr, CsBr	6:7:2:16	Ba:Zn:In:S	4.1	3.0	68
NaGaIn <sub>2</sub> Se <sub>5</sub>	HTS	850		1:2:4:9	Na <sub>2</sub> Se:Ga:In:Se	2.1	2.00	69
Na <sub>2</sub> In <sub>2</sub> MS <sub>6</sub> (M=Si, Ge)	HTS	620		SR	Na <sub>2</sub> S:In:M:S	0.3/0.5	2.47/2.41	70
Na <sub>2</sub> Ga <sub>2</sub> MS <sub>6</sub> (M=Ge, Sn)	HTS	800		SR	Na <sub>2</sub> S:Ga:M:S	0.8/1.1	3.10/2.74	71
Ln <sub>4</sub> GaSbS <sub>9</sub> (Ln= Sm, Gd, Ho)	HTS	950		8:1:1:15	Ln:Ga:Sb:S	3.8/0.8/0.25	2.23/2.41/2.44	72
Li <sub>4</sub> CdSn <sub>2</sub> S <sub>7</sub>	HTS	800		SR	Li <sub>2</sub> S:Cd:Sn:S	1.5	2.59	73
Li <sub>2</sub> In <sub>2</sub> MS <sub>6</sub> (M=Si, Ge)	HTS	900		SR	Li <sub>2</sub> S:In <sub>2</sub> S <sub>3</sub> :MS <sub>2</sub>	1/3	3.61/3.45	74
Li <sub>2</sub> In <sub>2</sub> MSe <sub>6</sub> (M=Si, Ge)	HTS	850		SR	Li <sub>2</sub> Se:In <sub>2</sub> Se <sub>3</sub> :MSe <sub>2</sub>	1/3	2.54/2.30	74
LiGaGe <sub>2</sub> Se <sub>6</sub>	HTS	750		SR	Li:Ga:Ge:Se	1	2.64	75
Pb <sub>4</sub> Ga <sub>4</sub> GeSe <sub>12</sub>	HTS	800		SR	Pb:Ga:Ge:Se	2	2.35	76
Sr <sub>6</sub> A <sub>4</sub> M <sub>4</sub> S <sub>16</sub> (A=Ag, Cu; M=Ge, Sn)	HTS	900		SR	SrS:A <sub>2</sub> S: MS <sub>2</sub> :SnS <sub>2</sub>	1.1/1.3/1.5/2.0	2.76/2.62/2.07/1.94	77
A <sub>2</sub> Ba <sub>7</sub> Sn <sub>4</sub> Q <sub>16</sub> (A = Li, Na)	HTS	870		SR	A:BaS:Sn:Q	0.5/0.1/0.2/0.4	2.5/2.3/2.1/1.75	78
Ba <sub>6</sub> Ag <sub>2.67</sub> Sn <sub>4.33</sub> S <sub>16</sub>	HTS	800	KBr	SR	Ba:Ag:Sn:S	6	1.58	79
Ba <sub>3</sub> CdSn <sub>2</sub> S <sub>8</sub>	HTS	950		SR	Ba:CdS:Sn:S	0.8	2.75	29
Sr <sub>3</sub> MnSn <sub>2</sub> S <sub>8</sub>	HTS	900		SR	SrS:Mn:Sn:S	1	3.02	80
Rb <sub>10</sub> Zn <sub>4</sub> Sn <sub>4</sub> S <sub>17</sub>	HTS	750	KI	SR	Rb <sub>2</sub> S <sub>3</sub> :Zn:Sn:S	3	3.59	81

Chalcohalides	Ba <sub>3</sub> AGa <sub>5</sub> Se <sub>10</sub> Cl <sub>2</sub> (A = Cs, Rb, K)	HTS	850	ACl, BaCl <sub>2</sub>	5:10:20:2:1	Ba:Ga:Se:ACl:BaCl <sub>2</sub>	100/20/10	2.08/2.05/2.04	82	
	Ba <sub>4</sub> MGa <sub>4</sub> Se <sub>10</sub> Cl <sub>2</sub> (M=Zn, Cd, Mn)	HTS	1000		SR	BaCl <sub>2</sub> :Ba:M:Ga:Se	59/52/30	3.08/2.93/2.78	83	
	NaBa <sub>4</sub> Ge <sub>3</sub> S <sub>10</sub> Cl	HTS	850			1:1:1	Ba:S:GeS <sub>2</sub> :NaCl	0.3	3.49	84
	Li[LiCs <sub>2</sub> Cl][Ga <sub>3</sub> S <sub>6</sub> ]	HTS	950	Ba		1.08:0.54:1.55:3.62:0.90	Li:Ba:Ga:S:CsCl	0.7	4.18	85
	[ABa <sub>2</sub> Cl][Ga <sub>4</sub> S <sub>8</sub> ] (A = Rb, Cs)	HTS	950			0.45:2.16:4.32:1.50	Ba:Ga:S:ACl	1/0.9	3.30/3.35	86
	[A <sub>3</sub> X][Ga <sub>3</sub> PS <sub>8</sub> ] (A=K, Rb; X=Cl, Br)	HTS	780	AX		1:3:1:10	AX:Ga:P:S	1.0/1.1/1.2/2.0	3.60/3.65/3.85/3.5	87
	Ba <sub>6</sub> Cs <sub>2</sub> In <sub>n</sub> Ga <sub>10-n</sub> Se <sub>20</sub> Cl <sub>4</sub> (n=1, 2)	HTS	600			SR	CsCl:BaCl <sub>2</sub> :Ba:In:Ga:Se	64/74	3.01/2.90	88
	Ba <sub>4</sub> M <sub>3</sub> Se <sub>9</sub> Cl <sub>2</sub> (M=Si, Ge)	HTS	950	BaCl <sub>2</sub>		1.2: 3: 3: 9	BaCl <sub>2</sub> :Ba:M:Se	1	1.76/1.89	89
	(K <sub>3</sub> I)[SmB <sub>12</sub> (GaS <sub>4</sub> ) <sub>3</sub> ]	HTS	950	KI		1:1:8:10	Sm:Ga:S:B	0.3*KDP	2.35	90
	(K <sub>3</sub> I)[InB <sub>12</sub> (InSe <sub>4</sub> ) <sub>3</sub> ]	HTS	950	KI		1:3:4	In:Se:B	0.1*KDP	1.97	91
	(K <sub>3</sub> X)[InB <sub>12</sub> (InS <sub>4</sub> ) <sub>3</sub> ] (X=Cl, Br, I)	HTS	950	KX		1:4:3	In:B:S	0.15~0.2	1.76/1.77/1.83	92
	(Cs <sub>3</sub> I)[InB <sub>12</sub> (InS <sub>4</sub> ) <sub>3</sub> ]	HTS	950	CsI		1:4:3	In:B:S	0.15~0.2	1.83	92
	(K <sub>3</sub> I)[IGdB <sub>12</sub> (GaS <sub>4</sub> ) <sub>3</sub> ]	HTS	950	KI		1:3:16:12	Gd:Ga:B:S	0.15~0.2	2.05	92
Li <sub>2</sub> C <sub>5</sub> G <sub>2</sub> S <sub>5</sub> (S <sub>2</sub> )Cl <sub>2</sub>	HTS	850			1:1:1:3	Li:CsCl:Ge:S	0.15	4.0	93	
Chalcoborates	LiBaB <sub>3</sub> S <sub>6</sub>	HTS	750		SR	Li <sub>2</sub> S:BaS:B:S	-	3.92	94	
	BaB <sub>2</sub> S <sub>4</sub>	HTS	700		SR	BaS:B:S	0.7	3.63	95	
	Ba <sub>3</sub> (BS <sub>3</sub> ) <sub>1.5</sub> (MS <sub>3</sub> ) <sub>0.5</sub> (M = Sb, Bi)	HTS	920		SR	Ba:B:M:S	-	3.01/2.43	96	
	Ba <sub>3</sub> (BS <sub>3</sub> )(SbS <sub>3</sub> )	HTS	820		SR	Ba:B:Sb:S	3	2.62	96	
	Ba <sub>3</sub> (BSe <sub>3</sub> )(SbSe <sub>3</sub> )	HTS	840		SR	Ba:B:Sb:Se	-	1.89	96	
	Zn <sub>0.2</sub> Ba <sub>2</sub> B <sub>2</sub> S <sub>5.2</sub>	HTS	900		SR	BaS:ZnS:B <sub>2</sub> S <sub>3</sub>	1.25*KDP		97	
Chalco phosphates	AP <sub>2</sub> Se <sub>6</sub> (A=K, Rb)	HTS	450		SR	A <sub>2</sub> Se:P <sub>2</sub> Se <sub>5</sub>	20/-	2.09/2.32	98,99	
	Cs <sub>2</sub> P <sub>5</sub> Se <sub>12</sub>	HTS	400		1:2.5:4	Cs <sub>2</sub> Se:P:Se	1	2.17	100	
	AZrPSe <sub>6</sub> (A=K, Rb, Cs)	HTS	900		2: 1:2:6	Cs <sub>2</sub> Se <sub>2</sub> :Zr:P <sub>2</sub> Se <sub>5</sub> :Se	45	2.00	101	
	Cs <sub>2</sub> BiP <sub>4</sub> Se <sub>12</sub>	HTS	850		2.5:1:4:9.5	Cs <sub>2</sub> Se:Bi:P:Se	6	1.85	102	
	ANb <sub>2</sub> PSe <sub>10</sub> (A = Cs, K, Rb)	HTS	600		0.5:0.25: 0.5:1	A <sub>2</sub> Se:Nb:P <sub>2</sub> Se <sub>5</sub> :Se	0.3	1.19/1.15/1.07	103	
	A <sub>4</sub> GeP <sub>4</sub> Q <sub>12</sub> (A = Cs, K, Rb)	HTS	500		SR	A <sub>2</sub> Se:Ge:P:Q	30/9/5	2.0/2.1/3.0/3.1	104	
	AgZnPS <sub>4</sub> (A = Ag, Li, Cu)	HTS	500		SR	A <sub>2</sub> S:ZnS:P <sub>2</sub> S <sub>5</sub>	1.8/0.8/3	2.76/3.38/3.0	105-107	
	CuHgPS <sub>4</sub>	HTS	900		SR	Cu:HgS:P:S	6.5	2.03	108	
	ACd <sub>3</sub> PS <sub>6</sub> (A = Ag, Li)	HTS	650		SR	A:Cd:P:S	0.45/0.8	2.56/2.97	109,110	
	CuCd <sub>3</sub> PS <sub>6</sub>	HTS	950		1: 3: 1	Cu <sub>2</sub> S:CdS:P <sub>2</sub> S <sub>5</sub>	0.9	2.24	111	

**Table S2.** The summary of recently developed pnictide IR NLO materials. high temperature solid state in sealed system (HTS), temperature (T), stoichiometric ratios (SR), second harmonic generation response (SHG), band gap ( $E_g$ ).

	Compounds	method	T (°C)	flux	Molar ratio	raw materials	SHG (*AGS)	$E_g$ (eV)	Ref.
Pnictides	CdSiP <sub>2</sub>	HTS	1180		SR	Cd:Si:P	6.5	2.20	112
	Ba <sub>2</sub> Si <sub>3</sub> P <sub>6</sub>	HTS	950		SR	Ba:Si:P	1.4	1.88	113
	MnSiP <sub>2</sub>	HTS	1050		1:1:2.01	Mn:Si:P	2.5	1.50	114
	MgSiP <sub>2</sub>	HTS	1150		SR	Mg:Si:P	5.3	2.24	115
	Ba <sub>4</sub> AgGa <sub>5</sub> P <sub>8</sub>	HTS	900	Pb	3:1:1:5:25	Ba:Ga:Ag:P:Pb	5.5	1.38	116
	BaGe <sub>2</sub> P <sub>2</sub>	HTS	950		SR	Ba:Ge:P	3	1.32	117
	Zn <sub>3</sub> PI <sub>3</sub>	HTS	600		1:4	Zn <sub>3</sub> P <sub>2</sub> :ZnI <sub>2</sub>	2.7	2.85	118
	Cd <sub>3</sub> PI <sub>3</sub>	HTS	700		1:5	Cd <sub>3</sub> P <sub>2</sub> :CdI <sub>2</sub>	3.5	2.44	118

**Table S3.** The summary of recently developed oxide IR NLO materials. solid-state reaction techniques in open system (SS), hydrothermal reactions (H), solvothermal reaction (ST), temperature (T), second haremomic generation response (SHG), band gap ( $E_g$ ).

Compounds	Method	T (°C)	Solution	Molar ratio	Raw materials	SHG	$E_g$ (eV)	Ref.
Pb <sub>17</sub> O <sub>8</sub> Cl <sub>18</sub>	SS	430		SR	PbO:PbCl <sub>2</sub>	2*AGS	3.44	119
Pb <sub>13</sub> O <sub>6</sub> Cl <sub>4</sub> Br <sub>10</sub>	SS	480		2:1:3	PbO:PbCl <sub>2</sub> :PbBr <sub>2</sub>	0.6*AGS	3.05	120
Pb <sub>13</sub> O <sub>6</sub> Cl <sub>7</sub> Br <sub>7</sub>	SS	460		1:1:1	PbO:PbCl <sub>2</sub> :PbBr <sub>2</sub>	0.8*AGS	3.13	120
Pb <sub>13</sub> O <sub>6</sub> Cl <sub>9</sub> Br <sub>5</sub>	SS	410		1:2:1	PbO:PbCl <sub>2</sub> :PbBr <sub>2</sub>	0.9*AGS	3.21	120
Pb <sub>18</sub> O <sub>8</sub> Cl <sub>15</sub> I <sub>5</sub>	SS	400		2:1:1	PbCl <sub>2</sub> :PbO:PbI <sub>2</sub>	2*AGS	2.82	121
Li <sub>3</sub> VO <sub>4</sub>	SS	680		SR	Li <sub>2</sub> CO <sub>3</sub> :V <sub>2</sub> O <sub>5</sub>	1.6*AGS	4.27	122
A <sub>2</sub> LiVO <sub>4</sub> (A = Rb,Cs)	SS	620		1:2.5:1	Li <sub>2</sub> CO <sub>3</sub> :A <sub>2</sub> CO <sub>3</sub> :V <sub>2</sub> O <sub>5</sub>	4/5*KDP	3.7/3.8	123
La <sub>3</sub> SnGa <sub>5</sub> O <sub>14</sub>	SS	1000		SR	La <sub>2</sub> O <sub>3</sub> :Ga <sub>2</sub> O <sub>3</sub> :SnO <sub>2</sub>	10*KDP	4.60	124
Li <sub>2</sub> ZrTeO <sub>6</sub>	SS	600		SR	Li <sub>2</sub> CO <sub>3</sub> :ZrO <sub>2</sub> :TeO <sub>2</sub>	2.5*KDP	4.06	125
Rb <sub>4</sub> Li <sub>2</sub> TiOGe <sub>4</sub> O <sub>12</sub>	SS	650		SR	Rb <sub>2</sub> CO <sub>3</sub> :Li <sub>2</sub> CO <sub>3</sub> :TiO <sub>2</sub> :GeO <sub>2</sub>	2*KDP	4.4	126
Li <sub>2</sub> K <sub>4</sub> TiOGe <sub>4</sub> O <sub>12</sub>	SS	700		SR	Li <sub>2</sub> CO <sub>3</sub> :K <sub>2</sub> CO <sub>3</sub> :TiO <sub>2</sub> :GeO <sub>2</sub>	2*KDP	4.43	127
K[VO <sub>2</sub> (IO <sub>3</sub> ) <sub>2</sub> ]	H	180	H <sub>2</sub> O	0.51:1.02:0.51	V <sub>2</sub> O <sub>5</sub> :KIO <sub>4</sub> :I <sub>2</sub> O <sub>5</sub>	3.6*KDP	2.0	128
A <sub>2</sub> BiI <sub>5</sub> O <sub>15</sub> (A = K, Rb)	H	230	H <sub>2</sub> O	6:6:1:6	AlO <sub>4</sub> :ACl:Bi <sub>2</sub> O <sub>3</sub> :H <sub>3</sub> IO <sub>6</sub>	3*KDP	3.50/3.53	129
Li <sub>2</sub> Ge(IO <sub>3</sub> ) <sub>6</sub>	H	230	H <sub>2</sub> O	2:0.5:5	LiCl:GeO <sub>2</sub> :HIO <sub>3</sub>	20*KDP	3.60	130
A <sub>2</sub> Ge(IO <sub>3</sub> ) <sub>6</sub> (A=K,Rb,Cs)	H	230	H <sub>2</sub> O	2:1:6	ACl:GeO <sub>2</sub> :HIO <sub>3</sub>	-	3.22/3.26/3.32	130
A <sub>2</sub> Sn(IO <sub>3</sub> ) <sub>6</sub> (A=Li,Na)	H	230	H <sub>2</sub> O	2.50:6.25:3.38	A <sub>2</sub> CO <sub>3</sub> :SnCl <sub>2</sub> :HIO <sub>3</sub>	400*SiO <sub>2</sub>	3.9/4.0	131
A <sub>2</sub> Sn(IO <sub>3</sub> ) <sub>6</sub> (A=K, Rb,Cs)	H	230	H <sub>2</sub> O	1.25:7.25:6.25	A <sub>2</sub> CO <sub>3</sub> :SnCl <sub>2</sub> :HIO <sub>3</sub>	-	4.0/4.1/4.1	131
A <sub>2</sub> Ti(IO <sub>3</sub> ) <sub>6</sub> (A=Li, Na)	H	230	H <sub>2</sub> O	1.7:1.3:17	A <sub>2</sub> CO <sub>3</sub> :TiO <sub>2</sub> :HIO <sub>3</sub>	500/400*SiO <sub>2</sub>	3.3	132
ABi <sub>2</sub> (IO <sub>3</sub> ) <sub>2</sub> F <sub>3</sub> (A= K,Rb,Cs)	H	210	HF+ H <sub>2</sub> O	2:0.5:2	AF:Bi <sub>2</sub> O <sub>3</sub> :H <sub>3</sub> IO <sub>6</sub>	12/9.5/7.5*KDP	3.75/3.78/3.84	133
$\alpha$ -AgI <sub>3</sub> O <sub>8</sub>	H	230	H <sub>2</sub> O	0.05:8	AgNO <sub>3</sub> :I <sub>2</sub> O <sub>5</sub>	9*KDP	3.78	134
$\beta$ -AgI <sub>3</sub> O <sub>8</sub>	H	230	H <sub>2</sub> O	0.5:8	AgNO <sub>3</sub> :I <sub>2</sub> O <sub>5</sub>	8*KDP	3.59	134
RbMoO <sub>3</sub> (IO <sub>3</sub> )	H	180	H <sub>2</sub> O	0.72:1.44	MoO <sub>3</sub> :RbIO <sub>4</sub>	400*SiO <sub>2</sub>	3.1	135
CsMoO <sub>3</sub> (IO <sub>3</sub> )	H	180	H <sub>2</sub> O	0.63:1.26	MoO <sub>3</sub> :CsIO <sub>4</sub>	400*SiO <sub>2</sub>	3.4	135
BaNbO(IO <sub>3</sub> ) <sub>5</sub>	H	230	H <sub>2</sub> O	1:0.6:9	Ba(IO <sub>3</sub> ) <sub>2</sub> :Nb <sub>2</sub> O <sub>5</sub> :I <sub>2</sub> O <sub>5</sub>	14*KDP	3.64	136
Bi(IO <sub>3</sub> )F <sub>2</sub>	H	230	HF+HNO <sub>3</sub> +H <sub>2</sub> O	0.25:1.5	Bi(NO <sub>3</sub> ) <sub>3</sub> :I <sub>2</sub> O <sub>5</sub>	11.5*KDP	3.97	137
Bi <sub>2</sub> (IO <sub>4</sub> )(IO <sub>3</sub> ) <sub>3</sub>	H	215	H <sub>2</sub> O	1:3:5	Bi(NO <sub>3</sub> ) <sub>3</sub> :HIO <sub>3</sub> :HNO <sub>3</sub>	5*KDP	3.3	138
BiO(IO <sub>3</sub> )	H	200	HNO <sub>3</sub>	1:1	Bi(NO <sub>3</sub> ) <sub>3</sub> :5H <sub>2</sub> O:HIO <sub>3</sub>	12.5*KDP	3.3	139
CsVO <sub>2</sub> F(IO <sub>3</sub> )	H	160	HF+ H <sub>2</sub> O	0.75:1.2:0.70	V <sub>2</sub> O <sub>5</sub> :Cs <sub>2</sub> CO <sub>3</sub> :I <sub>2</sub> O <sub>5</sub>	11*DKP	2.36	140
LiMg(IO <sub>3</sub> ) <sub>3</sub>	H	230	HCl+ H <sub>2</sub> O	6:1:2	LiCl:MgCl <sub>2</sub> :I <sub>2</sub> O <sub>5</sub>	1.5*AGS	4.34	141
RbIO <sub>3</sub>	H	230	H <sub>2</sub> O	1:2	Rb <sub>2</sub> CO <sub>3</sub> :HIO <sub>3</sub>	20*KDP	4.00	142
RbIO <sub>2</sub> F <sub>2</sub>	H	230	HF+H <sub>2</sub> O	3	RbIO <sub>3</sub>	4*KDP	4.20	142
Tl(VO) <sub>2</sub> O <sub>2</sub> (IO <sub>3</sub> ) <sub>3</sub>	H	200	H <sub>2</sub> O	0.5:0.125:2	TlI:V <sub>2</sub> O <sub>5</sub> :I <sub>2</sub> O <sub>5</sub>	12*KDP	2.37	143
Zn(IO <sub>3</sub> ) <sub>2</sub>	ST	60	nitric acid	-	Zn(NO <sub>3</sub> ) <sub>2</sub> :HgCl <sub>2</sub> :LiIO <sub>3</sub> :HIO <sub>3</sub>	2.7*KDP	5.10	144
NaI <sub>3</sub> O <sub>8</sub>	ST	60	nitric acid	-	NaIO <sub>3</sub>	5*KDP	-	145
NaYI <sub>4</sub> O <sub>12</sub>	H	220	H <sub>2</sub> O	2:1:22.7	Na <sub>2</sub> CO <sub>3</sub> :Y <sub>2</sub> O <sub>3</sub> :HIO	10*KDP	-	146
La(IO <sub>3</sub> ) <sub>3</sub>	H	220	H <sub>2</sub> O	1:2.84	La <sub>2</sub> O <sub>3</sub> :HIO <sub>3</sub>	10*KDP	-	146
Cs <sub>2</sub> I <sub>4</sub> O <sub>11</sub>	H	220	H <sub>2</sub> O	1:28.5:1.9	Cs <sub>2</sub> CO <sub>3</sub> :HIO <sub>3</sub> :Nb <sub>2</sub> O <sub>5</sub>	4.5*KDP	-	147
BaPd(IO <sub>3</sub> ) <sub>4</sub>	H	200	H <sub>2</sub> O	0.1:0.103:2	BaCO <sub>3</sub> :Pd(CH <sub>3</sub> COO) <sub>2</sub> :I <sub>2</sub> O <sub>5</sub>	4*KDP	1.97	148
RbAu(IO <sub>3</sub> ) <sub>4</sub>	H	200	H <sub>2</sub> O	0.03:0.05:4	Rb <sub>2</sub> CO <sub>3</sub> :Au(OH) <sub>3</sub> :I <sub>2</sub> O <sub>5</sub>	13.3*KDP	2.53	149

Oxides

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