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Rare-Earth Chalcogenides: A Large Family of Triangular Lattice Spin Liquid Candidates *

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Frustrated quantum magnets are expected to host many exotic quantum spin states like quantum spin liquid (QSL), and have attracted numerous interest in modern condensed matter physics. The discovery of the triangular lattice spin liquid candidate YbMgGaO_4 stimulated an increasing attention on the rare-earth-based frustrated magnets with strong spin-orbit coupling. Here we report the synthesis and characterization of a large family of rare-earth chalcogenides $A\text{ReCh}_2$ (A = alkali or monovalent ions, Re = rare earth, Ch = O, S, Se). The family compounds share the same structure ($\text{R}\bar{3}m$) as YbMgGaO_4 , and antiferromagnetically coupled rare-earth ions form perfect triangular layers that are well separated along the c -axis. Specific heat and magnetic susceptibility measurements on NaYbO_2 , NaYbS_2 and NaYbSe_2 single crystals and polycrystals, reveal no structural or magnetic transition down to 50 mK. The family, having the simplest structure and chemical formula among the known QSL candidates, removes the issue on possible exchange disorders in YbMgGaO_4 . More excitingly, the rich diversity of the family members allows tunable charge gaps, variable exchange coupling, and many other advantages. This makes the family an ideal platform for fundamental research of QSLs and its promising applications.

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The concept of quantum spin liquids (QSLs) was originally proposed by P. W. Anderson theoretically over 40 years ago.^[1] It describes a highly entangled quantum state for spin degrees of freedom and was initially constructed with a superposition of spin singlets on the triangular antiferromagnet, so-called resonating-valence-bond state.^[1] Later on, the possible connection between QSLs and high-temperature superconductivity was theoretically established through doping a QSL Mott insulator.^[2] Although the underlying mechanism for the high-temperature superconductivity has not yet come into a consensus, our understanding of QSLs has greatly improved, both from exactly solvable models^[3,4] and several classification schemes.^[4,5] On the experimental side, various frustrated magnetic materials, particularly the triangular-lattice-based antiferromagnets, were considered to be the most promising systems to realize QSLs.^[6] So far, a number of compounds have been reported to host QSLs. Among them, the well-known ones include herbertsmithite and its derived compounds,^[7–14] and triangular organics.^[15–19] The magnetic ions in most of these compounds are $3d$ transition metal ions Cu^{2+} with $S = 1/2$, which may be

crucial to enhance quantum fluctuations.

Quite recently, frustrated materials with magnetic rare-earth ions are proposed to be promising QSL candidates.^[20] These include the well-known pyrochlore ice materials,^[21–30] the kagome magnet,^[31,32] and the triangular lattice magnets.^[33–47] The local degree of freedom for the rare-earth ions that contain an odd number of $4f$ electrons (excluding Gd^{3+}) is a Kramers doublet and can be mapped to an effective spin $S = 1/2$ degree of freedom. This effective-spin local moment is protected by time reversal symmetry and the point group symmetry. In many cases the non-Kramers rare-earth ions can be taken as effective spin $S = 1/2$ local moments at low temperatures, though lacking the protection from time reversal symmetry.^[26,30,47–49] The spin-orbit-entangled nature of the rare-earth local moments often brings highly anisotropic spin models that have never been constructed and studied before.^[26,45–49] Thus, the rare-earth-based magnets play an important role in the exploration of novel spin models and the exotic magnetic states on various lattices. Indeed, QSL behaviors and multipolar phases have been proposed for various rare-earth compounds.^[21–30,32,46,47]

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The recent discovery of the triangular lattice magnet YbMgGaO₄ has invoked a further interest in the search of spin liquids with strong spin-orbit coupling.^[33,34,44,45,50–54] The compound has a space group symmetry of $R\bar{3}m$, and the Yb³⁺ ions form a flat and perfect triangular lattice.^[33,34] The availability of high-quality single crystals allows extensive and careful studies of magnetic properties using neutron scattering,^[36–39] muon spin relaxation (muSR),^[35] electron spin resonance (ESR)^[34] etc. These studies point to a possible gapless U(1) QSL ground state.^[33–37,39,40,52,53] On the other hand, some experiments and theoretical arguments raised the issue on Ga/Mg disorder, which was suggested to be responsible for the disordered spin state and/or QSL stability.^[33,37,39,54–56] The small exchange coupling allows an easy tunability of the spin state with a laboratory magnetic field.^[33,37,39,44] Meanwhile, it also requires that most experiments must be carried out at ultralow temperatures. In some cases this could be an obstacle for in-depth studies and possible applications.

As mentioned above, there is a long list of rare-

earth magnets.^[20,45,47] Then the question is if one can find out some interesting compounds or systems with larger exchange couplings and without disorder. This is the purpose of this work. We systematically synthesized the rare-earth chalcogenides AReCh₂ (A = alkali or monovalent ions, Re = rare earth, Ch = O, S, Se) with a delafossite structure. We carried out the structural and thermodynamic characterizations of these compounds. The compounds have a high symmetry of $R\bar{3}m$ and perfect spin triangular layers. The magnetic measurements indicate that spins are anti-ferromagnetically coupled in all the compounds with a range of Curie–Weiss temperatures. For the representative NaYbCh₂ (Ch = O, S, Se) samples, no magnetic ordering or transition is observed in the specific heat and susceptibility measurements down to 50 mK. Thus, this is a large family of QSL candidates with the simplest structure and chemical formula so far. Its crystal structure naturally removes the issue on Ga/Mg disorder proposed for YbMgGaO₄. The diversity of the large family makes it an ideal playground for studying the QSL physics and exploring its promising applications.

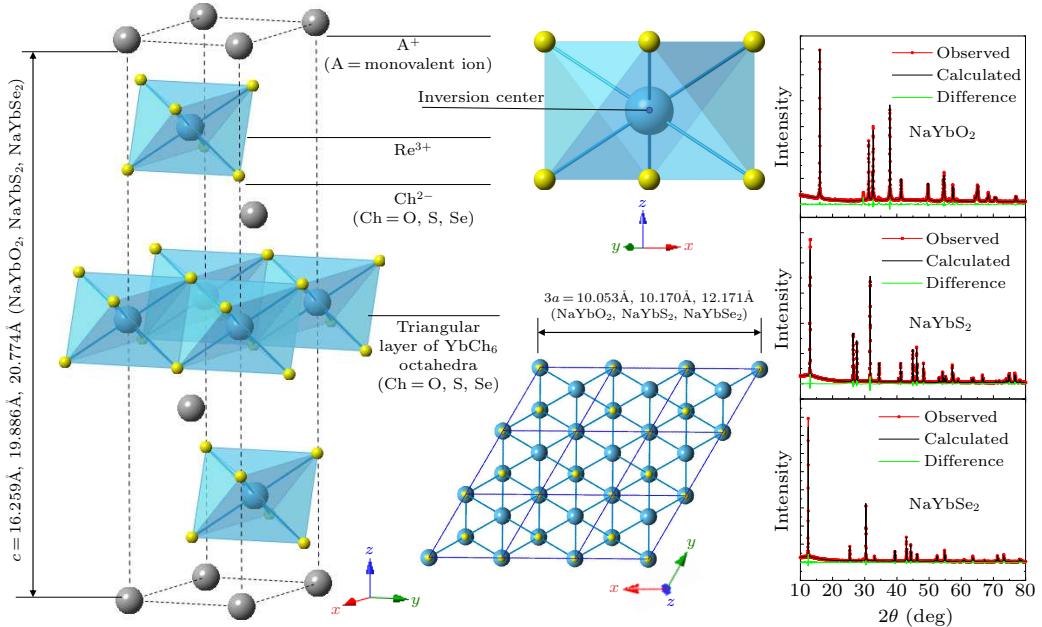


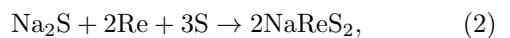
Fig. 1. The general crystal structure of rare-earth chalcogenides and the powder diffraction patterns and Rietveld refinements for NaYbCh₂ (Ch = O, S, Se).

The polycrystals of NaReO₂ (Re = Yb, Lu) were synthesized by the method of solid-state reaction under high temperatures:



Na₂CO₃ and Re₂O₃ powders were mixed in a dry process (mixing molar ratio: Na₂O:Yb₂O₃ = 2.5:1) and shaped into a pellet by isostatic pressing (50 MPa, 2 min). Shaped samples were heated at 900 °C for 9 hours. After the heating, the samples were ground

and washed with distilled water and ethanol, lastly dried in air at room temperature for 48 hours. The polycrystals of NaReS₂ (Re = La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu) were synthesized by the method of solid-state reaction under high temperature:



where Re = Lu, Se, Tm, Er, Ho, Dy, Tb, Gd, Eu, Sm, Nd, Pr, Ce, La. The Na₂S, Re and S powder was

mixed in Ar environment glove box. The mixed powders were placed in a graphite crucible and vacuum packaged with quartz tube. Packaged samples were heated at 850 °C for 48 hours. After the heating, the sample were powder and washed with distilled water and then dried in air at 50 °C for 6 hours. The synthesis of polycrystals of NaReSe₂ (Re = Er, Yb, Lu) were similar with the synthesis of powders of NaReS₂. The temperature was adjusted to 900 °C.

We have also successfully grown high quality NaYbSe₂ single crystals. The growth conditions of single crystals are more rigorous than NaYbSe₂ polycrystals. Na₂Se, Yb and Se powders were mixed in Ar environment glove box (mixing molar ratio: Na₂Se:Yb:Se = 1:2:12). The mixed powder was placed in special quartz tube that can withstand higher pressure. Packaged samples were heated at 1000 °C for 48 hours. After the heating, we can observe 2–3 mm size single crystals. We made a simple resistance check for the crystals and the resistance is overrange and thus NaYbSe₂ is confirmed to be a good insulator.

Powder XRD profiles were measured with Bruker-D8 by step scanning. The TOPAS program was used

for Rietveld crystal structure refinements. The temperature dependence of magnetic susceptibility from 1.8 K to 300 K was measured with a SQUID magnetometer (Quantum Design Magnetic Property Measurement System, MPMS) under both ZFC and FC for all the samples with the brass sample holder. The AC susceptibility measurements from 50 mK to 4 K were performed using a dilution refrigeration system (DR). The polycrystalline sample was pressed into a thin plate and fixed on a sample holder with GE vanish. The heat capacity measurements from 2 K to 30 K were performed using PPMS (Quantum Design Physical Property Measurement System) and DR was employed for the measurements from 50 mK to 4 K. The plate sample was mounted on a sample holder with N grease for a better thermal contact.

Table 1. Parameters extracted from Curie–Weiss fitting for NaYbCh₂ (Ch = O, S, Se).

Ch	Space group	C	Θ_{CW} (K)	μ_{eff} (μ_B)
O	$R\bar{3}m$	1.9448	−77.35	3.94
S	$R\bar{3}m$	2.6166	−40.98	4.57
Se	$R\bar{3}m$	2.2550	−59.84	4.24

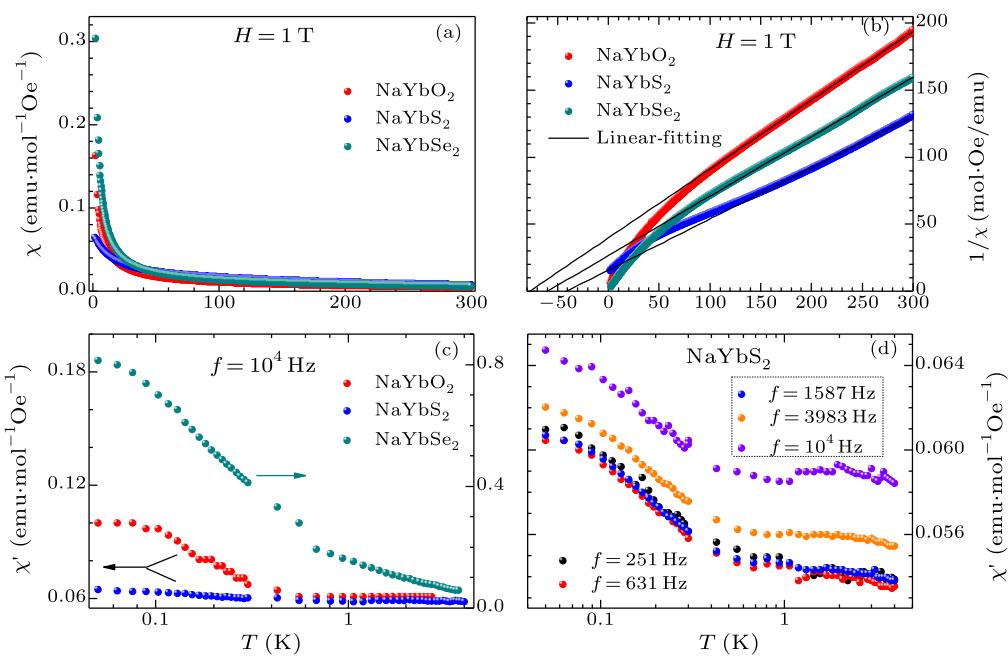


Fig. 2. The DC [(a), (b)] and AC [(c), (d)] magnetic susceptibilities of polycrystalline NaYbCh₂ (Ch = O, S, Se).

In Fig. 1, we depict the crystal structure of the rare-earth chalcogenides and the Rietveld refinements for three representative samples NaYbO₂, NaYbS₂ and NaYbSe₂, where the detailed structural information extracted from the refinements can be found in the Supplementary Materials. The system has an $R\bar{3}m$ space group symmetry, and the magnetic ions form flat triangular layers that are well separated. The ReCh₆ octahedra are connected in an edge-

sharing fashion. The local crystal-field environment around the magnetic ions is exactly analogous to the case of YbMgGaO₄. Therefore, one expects a similar crystal-field splitting scheme of the Yb³⁺ ions as the one in YbMgGaO₄. This means that a spin-orbital-entangled effective spin $S = 1/2$ local moment should hold for our case. Similar to YbMgGaO₄, the anti-symmetric Dzyaloshinskii-Moriya interaction is prohibited by the inversion symmetry of the system.

The Mg/Ga disorder in the non-magnetic layers of YbMgGaO_4 has been extensively discussed and is still under debate. Whether or how much this non-magnetic disorder impacts on the Yb magnetic properties is unclear in this stage. In some experiments and theoretical calculations, the disorder was considered to play a dominant role in contributing to the low-energy excitations. As a comparison, there is no such disorder in this family of rare-earth chalcogenides, due to the structural simplicity. The issue on disorder is completely removed for this family of materials. We further made the analysis of element ratio (see Supplementary Materials), which is close to the nominal ratio. This rules out the possibility of the disorder caused by element deficiency. If one still concerns about the active monovalent ions like Na^+ and K^+ , he will have plenty of choices of the heavy monovalent ones such as Rb^+ , Cs^+ , Cu^+ and Ag^+ .

For the selected sub-family NaYbCh_2 ($\text{Ch} = \text{O}, \text{S}, \text{Se}$), we measured the DC magnetic susceptibility in the range of 2–300 K and the AC susceptibility from 50 mK to 4 K. The results are presented in Fig. 2. The Curie–Weiss fitting was made from 150 K to 300 K according to the crystal-field splitting in YbMgGaO_4 and the fitting results are summarized in Table 1. Considering the small interaction energy scale of rare-earth moments, this fitting range may not be quite sufficient to characterize the low-energy

magnetic physics of the system. A lower fitting range may be required in the future. Nevertheless, the negative Curie–Weiss temperatures suggest an antiferromagnetic coupling in all the samples. Excitingly, the Curie–Weiss temperatures are much larger than that of YbMgGaO_4 because of the smaller distances between nearest-neighbor Yb^{3+} in the rare-earth chalcogenides. The AC susceptibility for the three samples shows no sign of long-range magnetic ordering. The measurements under various frequencies further confirms that there is no spin freezing either. Interestingly, the susceptibility saturation in the zero temperature limit is clearly observed for all the three samples. This should be one of the consequences caused by strong spin-orbit coupling rather than a sign of finite density of spin excitations. Under the strong spin-orbit coupling, the total magnetization is not a good quantum number and cannot be used to label the many-body eigenstate. The many-body eigenstate would be a mixture of states with different total magnetizations. The magnetic susceptibility would always be a constant. We also find that the low-temperature susceptibility of NaYbSe_2 is obviously larger than that of the other two compounds. In fact, the distance between nearest neighbor Yb^{3+} ions in NaYbSe_2 is larger, while its Curie–Weiss temperatures and the moment obtained from the Curie–Weiss fitting look comparable to the other two.

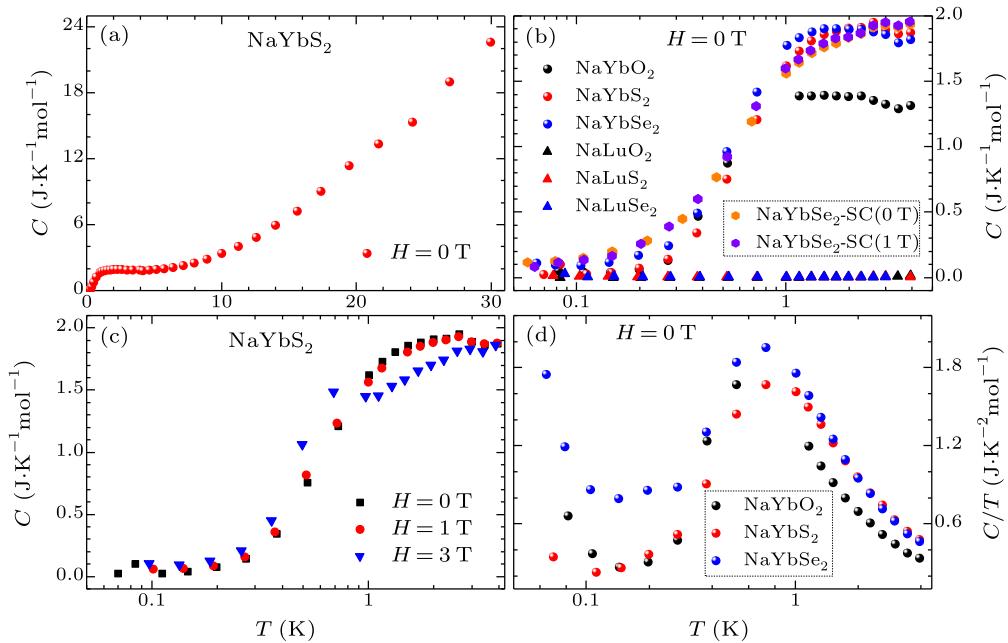


Fig. 3. Specific heat measurements on NaYbCh_2 ($\text{Ch} = \text{O}, \text{S}, \text{Se}$). Single crystal (SC) NaYbSe_2 and polycrystalline NaYbO_2 and NaYbS_2 were used in the measurements.

Our specific heat results are shown in Fig. 3. There is no obvious transition down to 50 mK in these compounds, and it is consistent with the conclusion from the magnetic susceptibility data. We observed no ap-

parent change or shift with applying a magnetic field up to 3 T (see Fig. 3(c)). On the other hand, an upturn is observed below 100 mK in the $C/T-T$ plot (see Fig. 3(d)). This may arise from the nuclear Schottky

anomaly due to the nuclear spins. The upturn makes it difficult to obtain the intrinsic trend of the specific heat below 100 mK and to conclude whether the system is a gapless or gapped QSL. Thus, more detailed magnetic and thermodynamic experiments are required in the future. The broad peak around 1 K in the $C/T-T$ plot is considered to be a consequence of preserving entropy. Here we point out that there is no obvious disorder in the present case and we still do not observe any long-range magnetic ordering or freezing that points to a possible QSL ground state. This means that the rare-earth triangular system, including rare-earth chalcogenides reported here and YbMgGaO_4 , intrinsically hosts the QSL state that is not stabilized by or even originated from the Ga/Mg charge disorder.

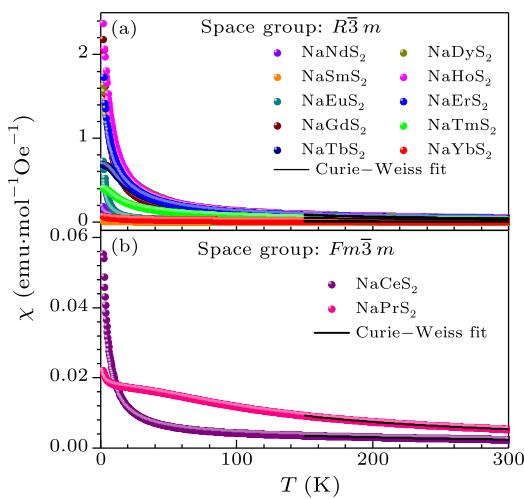


Fig. 4. Magnetic susceptibility of NaReS_2 ($\text{Re}=\text{Ce–Yb}$) in the range of 2 K to 300 K.

The measurements discussed above are based on the sub-family NaYbCh_2 ($\text{Ch}=\text{O, S, Se}$). Towards a comprehensive view of the large family, we fixed Na and S, and systematically synthesized the other sub-family NaReS_2 ($\text{Re}=\text{La–Lu}$). The Rietveld refinements for all the fourteen compounds have been made and the detailed structural parameters can be found in the Supplementary Materials. The family members from Nd to Lu preserve the high lattice symmetry of $R\bar{3}m$. But the three end members, NaLaS_2 , NaCeS_2 and NaPrS_2 show a cubic structure with the space group symmetry of $Fm\bar{3}m$. Clearly this is caused by the larger ionic radii of La, Ce and Pr. The magnetic susceptibility measurements from 2 K to 300 K have been carried out on the sub-family and the results are presented in Fig. 4. No obvious magnetic transition is observed in all the compounds with the $R\bar{3}m$ symmetry. Similar to the $R\bar{3}m$ brothers, the cubic NaCeS_2 also shows no sign of magnetic transition from 2 K to 300 K, as the perfect triangular lattice formed by the magnetic ions remains undistorted and the strong geometrical frustration is always there. In contrast, an anomaly appears around 10 K in the susceptibility of

NaPrS_2 ($Fm\bar{3}m$).

The Curie–Weiss fitting was performed from 150 K to 300 K, assuming a reasonably high crystal field splitting. The full results are presented in Table 2. We can see that the exchange couplings vary from sample to sample. In the other words, we have the opportunity to select the compounds with various exchange coupling. Beyond this, one can further tune the charge gaps of the family members by element substitution. The absorption spectra (see Supplementary Materials) indicate that the charge gaps are roughly 4.5 eV, 2.7 eV and 1.9 eV for NaYbO_2 , NaYbS_2 , and NaYbSe_2 , respectively. The variable and small charge gaps may allow the system to access a Mott–metal transition by applying doping or pressures. Such a possibility opens up the interesting direction of Mott transitions out of a QSL.^[57] This transition was argued to be continuous by noticing that the Landau damping term scales like a mass term for the bosonic charge and then identifying the transition as an usual superfluid–Mott transition.^[57] Thus, these exciting advantages stem from the rich diversity of the family. In fact, we have made a careful literature research and found that most of the family members have the high symmetry of $R\bar{3}m$ and hence are potential QSL materials (see Supplementary Materials). This suggests that the family is an ideal playground, on which we can tune the basic material parameters or exchange coupling to explore the QSL physics and develop its possible applications.

Table 2. Parameters extracted from the Curie–Weiss fitting for NaReS_2 ($\text{Re}=\text{Ce–Yb}$).

Re	Space group	C	θ_{CW} (K)	μ_{eff} (Obs.)	μ_{eff} (Cal.)
Ce	$Fm\bar{3}m$	1.1145	-164.42	$2.99\mu_B$	$2.54\mu_B$
Pr	$Fm\bar{3}m$	1.91757	-57.48	$3.92\mu_B$	$3.58\mu_B$
Nd	$R\bar{3}m$	1.78198	-25.35	$3.77\mu_B$	$3.62\mu_B$
Sm	$R\bar{3}m$	0.71761	-343.05	$2.40\mu_B$	$0.84\mu_B$
Eu	$R\bar{3}m$	3.19082	-106.13	$5.05\mu_B$	$3.6\mu_B$
Gd	$R\bar{3}m$	8.5521	-1.98	$8.27\mu_B$	$7.94\mu_B$
Tb	$R\bar{3}m$	12.87953	-9.49	$10.15\mu_B$	$9.72\mu_B$
Dy	$R\bar{3}m$	14.55906	-9.39	$10.79\mu_B$	$10.63\mu_B$
Ho	$R\bar{3}m$	13.8468	-5.90	$10.52\mu_B$	$10.60\mu_B$
Er	$R\bar{3}m$	12.13713	-4.62	$9.85\mu_B$	$9.59\mu_B$
Tm	$R\bar{3}m$	7.40589	-3.83	$7.69\mu_B$	$7.57\mu_B$
Yb	$R\bar{3}m$	2.90963	-63.74	$4.82\mu_B$	$4.54\mu_B$

In summary, we have synthesized rare-earth chalcogenides $A\text{ReCh}_2$ that exhibit a delafossite structure, and made structural and thermodynamics characterizations. The family has a lattice symmetry $R\bar{3}m$, and the magnetic ions are antiferromagnetically coupled and form perfect triangular layers. The magnetic susceptibility and specific heat measurements down to 50 mK indicate no sign of long-range magnetic ordering or transition. The family removes the disorder issue raised in YbMgGaO_4 and also suggests the QSL physics is probably not from disorder. The unique advantages, such as various charge gaps and exchange coupling, suggest that the family may be an ideal platform for the further study of QSLs.

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Note added: Upon the completion of this work, we become aware of Ref. [58] that focused on NaYbS₂ and proposed it as a spin liquid candidate.

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