

Cesium fluorohydrogenate, Cs(FH)_{2,3}F

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Abstract

$\text{Cs}(\text{FH})_{2.3}\text{F}$ is a liquid salt exhibiting a low viscosity of 20.1 cP and a high conductivity of 86.3 mS cm^{-1} at $25 \text{ }^\circ\text{C}$, in spite of the relatively high melting point (16.9°C). The high density of 2.82 g cm^{-3} at the liquid state is due to the heavy atomic weight and small size of cesium atom compared to the organic cations of general ionic liquids. The infrared spectroscopy indicates that this salt contains $(\text{FH})_2\text{F}^-$ as a main anionic species. The other anionic species such as $(\text{FH})_3\text{F}^-$ found in the cases of other $\text{M}^+(\text{HF})_{2.3}\text{F}$ (M = a univalent organic cation) ionic liquid salts is not detected, suggesting its small abundance as well as the presence of neutral HF in the form of molecular and/or oligomers. The result of $^1\text{H-NMR}$ also suggests that the anions exchange HF between them. These observations coincide with the experimental result that $\text{Cs}(\text{FH})_{2.3}\text{F}$ acts as an acid against general ionic liquid fluorohydrogenates such as $\text{EMIm}(\text{FH})_{2.3}\text{F}$ to lose HF and give $\text{Cs}(\text{FH})_2\text{F}$ precipitate.

1. Introduction

It has been known that alkaline metal fluorides (MF, M = alkaline metal) form complex salts with HF [1,2]. These salts are composed of M^+ cations and fluorohydrogenate $((FH)_nF^-$, where n is an integer) anions. KF-HF system is used as an electrolyte for electrochemical synthesis of elemental fluorine and many studies have been made on its physical, chemical and electrochemical properties [3-5]. A similar application was also examined for CsF-HF system, although it was not applied to industrial electrolysis due to the high cost of CsF [2,6]. The advantage of CsF-HF system is its low melting point compared to KF-HF system which enables the electrolysis to be performed at lower temperatures to save energy and give broader choice of materials of electrolytic cells. According to the phase diagram [2], the CsF-HF system has a eutectic point below room temperature at the composition of CsF:HF = 3:7 (m.p. 16.9 °C), corresponding to the formula, $Cs(FH)_{2.3}F$.

Recently, room temperature ionic liquids have been widely studied as new functional electrolytes and solvents [7-16]. Their unique properties such as nonvolatility, nonflammability and wide liquid temperature ranges are attractive for various electrochemical and chemical applications. The authors have reported a series of room temperature ionic liquids composed of organic cations and $(FH)_nF^-$. EMIIm $(FH)_{2.3}F$ (EMIIm = 1-ethyl-3-methylimidazolium) was the first characterized in the series of dialkylimidazolium salts [17,18]. It was revealed that EMIIm $(FH)_{2.3}F$ was composed of EMIIm $^+$, $(FH)_2F^-$ and $(FH)_3F^-$ regarded as a mixed salt and exhibits a high conductivity of 100 mS cm^{-1} and a low viscosity of 4.9 cP. The effects of the side-chain [19] and the frame structure of cationic species [20] as well as the HF composition n in anionic species [21] on the physical properties were studied by using several methods. Transport

mechanism in RMIIm(FH)_{2,3}F (RMIIm = 1-alkyl-3-methylimidazolium) were investigated using pulsed-gradient spin echo NMR technique [22]. Structural characteristics of EMImFHF at both the liquid and solid states and RMIIm(FH)_{2,3}F in the liquid state were studied using X-ray diffraction techniques [23-25]. Some spectroscopic data are also available for TMA(FH)_nF (TMA = tetramethylammonium, $n = 3.0 - 5.0$) salts that show some HF dissociation pressures at ambient temperatures [26]. Cs(FH)_{2,3}F is also a potential electrolyte for electrochemical energy conversion devices like other fluorohydrogenate salts. Moreover, its use as a recyclable solvent and electrolyte for chemical and electrochemical syntheses is an attractive application. In spite of such potential interests, the properties of this complex salt were reported long time ago and the data available today is still not comprehensive. In the present study, some physical and structural properties of Cs(FH)_{2,3}F are reported and compared to those of the other fluorohydrogenate salts.

2. Results and discussion

Colorless liquid was obtained after the reaction of CsF and large excess of anhydrous HF. Along with the elimination of HF by pumping, the viscosity of the liquid was increased and colorless crystalline solid was formed in the liquid. This solid melted to become a homogeneous liquid again by further elimination of HF. The liquid slowly released HF to solidify again under dynamic vacuum at room temperature. The observation well agrees with the phase diagram of CsF-HF system given by Winsor and Cady many years ago (Fig. 1) [2]. According to the phase diagram, the solid phase of Cs(FH)₃F appears at the composition of Cs(FH)_{3,5}F at room temperature by elimination of HF from the HF-rich liquid of the CsF-HF system, and the solid disappears at the

composition corresponding to $\text{Cs}(\text{FH})_{2.7}\text{F}$ to give liquid again. Another solid phase of $\text{Cs}(\text{FH})_2\text{F}$ appears for $\text{Cs}(\text{FH})_{2.1}\text{F}$ by further elimination of HF after crossing the eutectic point at which the $\text{Cs}(\text{FH})_{2.3}\text{F}$ is obtained.

$\text{Cs}(\text{FH})_{2.3}\text{F}$ has a non-zero HF dissociation pressure at room temperature and slowly releases HF. Since nonvolatility with the absence of neutral molecules in the liquid is essential for room temperature ionic liquids, rigorously speaking, $\text{Cs}(\text{FH})_{2.3}\text{F}$ may not be classified into this category even without taking account the fact that all the ionic liquids by current definition contain organic cations and/or anions whereas this salt does not. However, the HF dissociation pressure of $\text{Cs}(\text{FH})_{2.3}\text{F}$ is less than a few Torr at room temperature according to a rough estimation using a Pirani gauge which is low enough for some practical applications at ambient conditions. In the case of EMImF-HF system, the EMIm(FH) $_n$ F salt is vacuum-stable at room temperature in the region of $n \leq 2.3$ [17-19, 21]. In other words, EMIm(FH) $_{2.3}$ F has the largest HF composition as a nonvolatile room temperature ionic liquid in EMImF-HF system. This magic number of 2.3 has been observed for various vacuum-stable fluorohydrogenate salts at room temperature using organic cations [19-20]. Although n at the eutectic HF composition of $\text{Cs}(\text{FH})_n\text{F}$ is also 2.3, physical meaning of this coincidence is not clear in this stage. According to the phase diagram of EMIm(FH) $_{2.3}$ F, the salt does not exhibit an eutectic point at this composition [21].

Some physical properties of $\text{Cs}(\text{FH})_{2.3}\text{F}$ are summarized in Table 1 with those of EMIm(FH) $_{2.3}$ F and BMIm(FH) $_{2.3}$ F [19] for comparison. The conductivity of $\text{Cs}(\text{FH})_{2.3}\text{F}$ is 86 mS cm^{-1} that is high compared to those of the other known room temperature ionic liquids, for example 14 mS cm^{-1} for EMImBF $_4$ [27] and 8.8 mS cm^{-1} for EMImN(SO $_2$ CF $_3$) $_2$, [28] and comparable to 100 mS cm^{-1} for EMIm(FH) $_{2.3}$ F. Generally,

fluorohydrogenate salts exhibit relatively low viscosities, such as 4.9 cP for EMIIm(FH)_{2,3}F [19]. The low viscosity increases mobility of the ionic species in the liquid and results in its high conductivity [18-21, 29]. However, the viscosity of Cs(FH)_{2,3}F is close to that of BMIm(FH)_{2,3}F of which the ionic conductivity is 33 mScm⁻¹. This is explained by the difference in sizes of the Cs and BMIm cations that causes a significant difference in the number of carriers per unit volume. As shown in Table 1, the molar conductivities of them are close to each other as a result of the similar viscosities of them. Higher ionic conductivity observed for the Cs salt is therefore given by the effective packing of charge carrier by the highly symmetric small cation. The large density value of 2.82 g cm⁻³ is one of the notable properties of Cs(FH)_{2,3}F and is due to the large atomic weight of Cs atom with a small volume compared to bulky organic cations generally used for room temperature ionic liquids.

Cs(FH)_{2,3}F is composed of Cs⁺ and (FH)_nF⁻ as in the case for other fluorohydrogenate salts. (FH)_nF⁻ anions in other salts have been characterized by several methods such as single-crystal X-ray diffraction, IR spectroscopy and theoretical calculations [17-21, 26, 30-38]. Abundance of the anionic species in fluorohydrogenate salts changes depending on the cationic species and HF composition *n*. EMIIm(FH)_{2,3}F which has a negligible HF dissociation pressure at room temperature (< 10⁻² Torr) contains two anionic species, (FH)₂F⁻ and (FH)₃F⁻, and does not contain detectable amount of free HF molecule [17-19]. On the other hand, Cs(FH)_{2,3}F has a non-zero HF dissociation pressure as mentioned above, suggesting the presence of free HF in the liquid although the abundance is expected to be small. In the IR spectrum of Cs(FH)_{2,3}F shown in Fig. 2, broad absorption bands observed at ~ 500, 1050, and 1800 are definitely assigned to (FH)₂F⁻ [17-21, 26, 33-36] that is regarded as a main anionic species in this salt. FHF⁻ is

known to exhibit a strong absorption band at $\sim 1250\text{ cm}^{-1}$ in IR spectrum, whereas an absorption band of $(\text{FH})_3\text{F}^-$ is often unclear but appears at $\sim 900\text{ cm}^{-1}$ [17-21, 26, 33-36]. In the IR spectrum of $\text{Cs}(\text{FH})_{2.3}\text{F}$, there is no band at 1250 cm^{-1} and FHF^- is concluded not to exist in this system. The absorption assigned to $(\text{FH})_3\text{F}^-$ ($\sim 900\text{ cm}^{-1}$) is hard to be identified clearly as a single peak in the spectrum due to the overlap with that from $(\text{FH})_2\text{F}^-$. Liquid anhydrous HF gives a broad band at $\sim 3440\text{ cm}^{-1}$ [39]. The weak band at $\sim 3300\text{ cm}^{-1}$ in the spectrum of $\text{Cs}(\text{FH})_{2.3}\text{F}$ which is not observed for the spectrum of $\text{EMIm}(\text{FH})_{2.3}\text{F}$ is reasonably ascribed to HF that is not bound to fluoride anion in the liquid. $(\text{FH})_3\text{F}^-$ might exist in $\text{Cs}(\text{FH})_{2.3}\text{F}$, but the abundance is probably smaller than that found for other fluorohydrogenates ionic liquids taking account the presence of HF not belonging to the anions.

Figure 3 (a) and (b) show the ^1H -NMR and ^{19}F -NMR spectra of neat $\text{Cs}(\text{FH})_{2.3}\text{F}$, respectively. Only one singlet peak assigned to the anionic species and HF present in the liquid discussed above was observed in both the spectra, indicating that the fast exchange of HF between the anions occurs in $\text{Cs}(\text{FH})_{2.3}\text{F}$. In the ^1H -NMR spectrum, the chemical shift for the proton is observed at 11.5 ppm. The chemical shift is 12 ppm for the anionic species in the neat $\text{EMIm}(\text{FH})_{2.3}\text{F}$. On the other hand, the chemical shift for the anionic species and HF in $\text{TMA}(\text{FH})_n\text{F}$ ($n = 3.5, 4.0, 4.5$ and 5.0) is observed in the range between 9.74 and 9.84 ppm. $\text{EMIm}(\text{FH})_{2.3}\text{F}$ contains only ionic species and does not contain free HF, whereas $\text{TMA}(\text{FH})_n\text{F}$ exhibits a higher HF dissociation pressure and contains a significant amount of free HF in addition to the anionic species, FHF^- , $(\text{FH})_2\text{F}^-$ and $(\text{FH})_3\text{F}^-$ [26]. The observed values of the ^1H chemical shifts suggest that the proton in $\text{Cs}(\text{FH})_{2.3}\text{F}$ is more weakly shielded than that in the $\text{TMA}(\text{FH})_n\text{F}$ melt but not as much as that in the $\text{EMIm}(\text{FH})_{2.3}\text{F}$ melt. This observation is explained by the average number of

fluorine atoms bound to a proton in the liquid which is two for the proton in the anion and one for that in the neutral HF. With the increase of free HF where proton is bound to only one fluorine atom and more shielded than that in the anion, the peak shift occurs to the higher magnetic field. The similar discussion can be made on the chemical shifts in the ^{19}F -NMR spectra of these fluorohydrogenates. In this case, fluorine nuclei in HF is more shielded by bound with fluoride anion. Thus the ^{19}F -NMR signal for $\text{EMIm}(\text{FH})_{2,3}\text{F}$ (-103 ppm) is observed at higher magnetic field than that for $\text{Cs}(\text{FH})_{2,3}\text{F}$ (-95.1 ppm).

The room temperature liquid-liquid reaction of $\text{Cs}(\text{FH})_{2,3}\text{F}$ and $\text{EMIm}(\text{FH})_{2,3}\text{F}$ gives white $\text{Cs}(\text{FH})_2\text{F}$ precipitate, indicating $\text{Cs}(\text{FH})_{2,3}\text{F}$ acts as an acid against $\text{EMIm}(\text{FH})_{2,3}\text{F}$ to lose HF.



In other words, a stronger Lewis base $\text{EMIm}(\text{FH})_{2,3}\text{F}$ substitutes a weak base $\text{Cs}(\text{FH})_2\text{F}$. This observation clearly demonstrates the higher acidity of fluorohydrogenate anion in $\text{Cs}(\text{FH})_{2,3}\text{F}$ than that in $\text{EMIm}(\text{FH})_{2,3}\text{F}$ as well as the stability of fluorohydrogenate anions in the latter against the dissociation of HF.

3. Experimental

3.1. General experimental procedure

Volatile materials were handled in a vacuum line constructed of SUS316 stainless steel and PFA (tetrafluoroethylene-perfluoroalkylvinylether copolymer). Nonvolatile materials were handled under a dry argon atmosphere in a glove box. A PFA reaction tube combined with a stainless steel valve was used as a reaction vessel. Anhydrous HF (Daikin Industries, purity > 99 %) was dried over K_2NiF_6 (Ozark-Mahoning) for several days prior to use. CsF (Wako chemicals, purity > 97 %) was well-dried under vacuum at 250 °C before use. IR spectrum was obtained by FTS-165 (BIO-RAD Laboratories). The

samples were sandwiched by a pair of AgCl windows fixed in a stainless airtight cell. The sample for NMR spectroscopy sealed in a FEP tube was placed in a glass NMR sample tube. ^1H -NMR measurement of the sample was performed using a Varian Mercury-300BB NMR spectrometer (300 MHz) and the obtained spectrum was referenced to tetramethylsilane (TMS). ^{19}F -NMR measurement of the sample was performed using a Varian Mercury-300BB NMR spectrometer (282 MHz) and the obtained spectrum was referenced to trichlorofluoromethane (CFCl_3). C_6D_6 was used as an external standard. Conductivity was measured by impedance technique with the aid of HZ-3000 electrochemical measurement system (Hokuto Denko). The cell for conductivity measurement was made of PFA and PTFE (polytetrafluoroethylene) with platinum disk electrodes and was calibrated by KCl standard aqueous solution. Viscosity measurement was performed using an Ostwald viscometer made of PFA. Density was measured by weighing the sample in a PFA vessel whose volume was calibrated by distilled water. Differential scanning calorimetric (DSC) analysis was performed on the sample in a nickel cell under nitrogen gas flow using a Shimadzu DSC-60. A scanning rate 10 K min^{-1} was used.

3.2. Synthesis of $\text{Cs}(\text{FH})_{2.3}\text{F}$

Large excess of anhydrous HF was condensed on CsF loaded in a PFA reaction vessel at liquid nitrogen temperature. During warming up the vessel to room temperature, the reaction proceeded and colorless liquid was obtained. *CAUTION*: This reaction is highly exothermic and sometimes vigorous. The liquid was evacuated at room temperature until the sample was frozen first and melted again. The composition of the obtained colorless liquid was identified as $\text{Cs}(\text{FH})_{2.3}\text{F}$ ($\text{CsF} : \text{HF} = 3 : 7$) from the weight change.

3.3. Reaction of $EMIm(FH)_{2,3}F$ and $Cs(FH)_{2,3}F$

Liquid-liquid mixing of $EMIm(FH)_{2,3}F$ and $Cs(FH)_{2,3}F$ was made at ambient conditions in a PFA reaction vessel under an inert atmosphere. A white solid was precipitated immediately after the mixing. The solid was recovered by filtration. Melting point measured by DSC was 322K.

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Figure captions

Fig. 1 Phase diagram of CsF-HF system. The figure was reproduced from the original appeared in the reference 2.

Fig. 2 IR spectrum of Cs(FH)_{2,3}F at room temperature.

Fig. 3 (a) ¹H- and (b) ¹⁹F-NMR spectra of Cs(FH)_{2,3}F at room temperature.

Table 1 Physical properties of Cs(FH)_{2,3}F and EMIm(FH)_{2,3}F at 25 °C.

Salt	m.w.	m.p. / °C	Density / g cm ⁻³	Viscosity /cP	Conductivity / mS cm ⁻¹	Molar conductivity / S cm ² mol ⁻¹
Cs(FH) _{2,3} F	197.9	16.9	2.82	20.1	86	6.0
EMIm(FH) _{2,3} F [19]	176	-65	1.13	4.9	100	15.6
BMIm(FH) _{2,3} F [19]	204	-	1.08	19.6	33	6.2

m.w.: molecular weight, m.p.: melting point.

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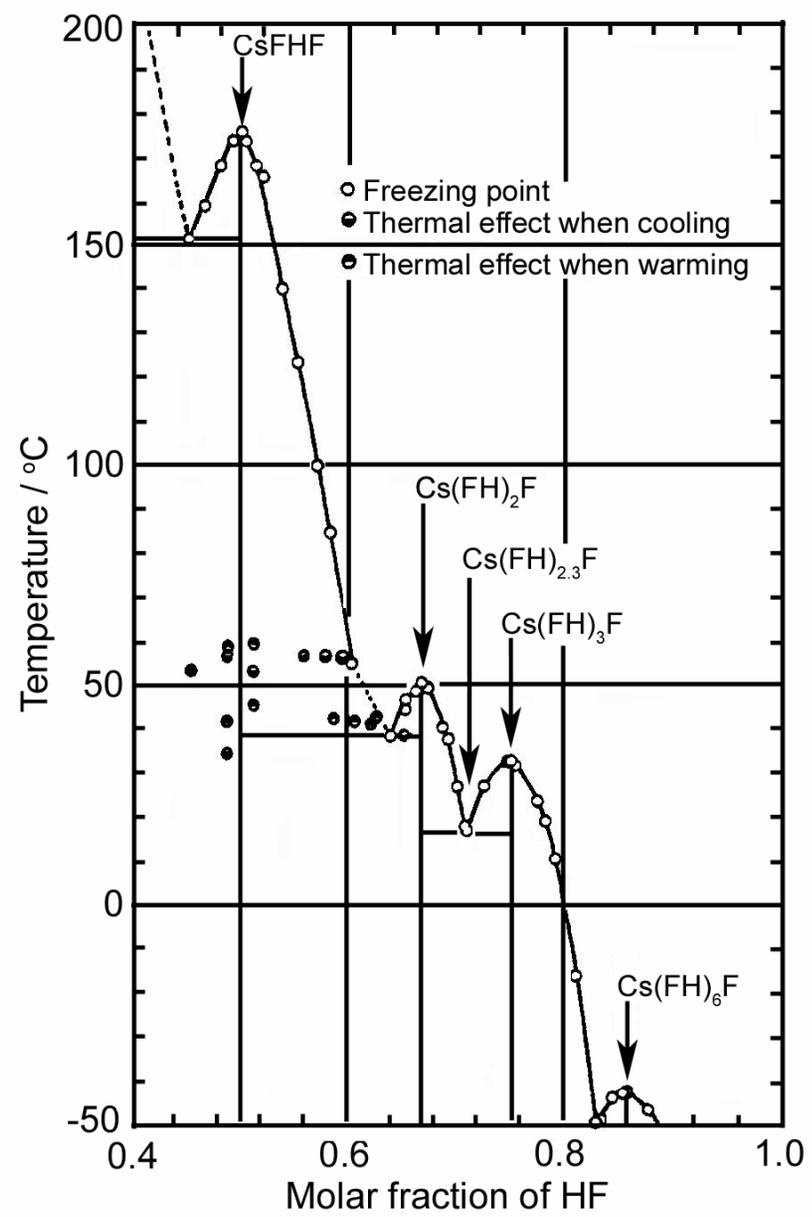


Fig. 1
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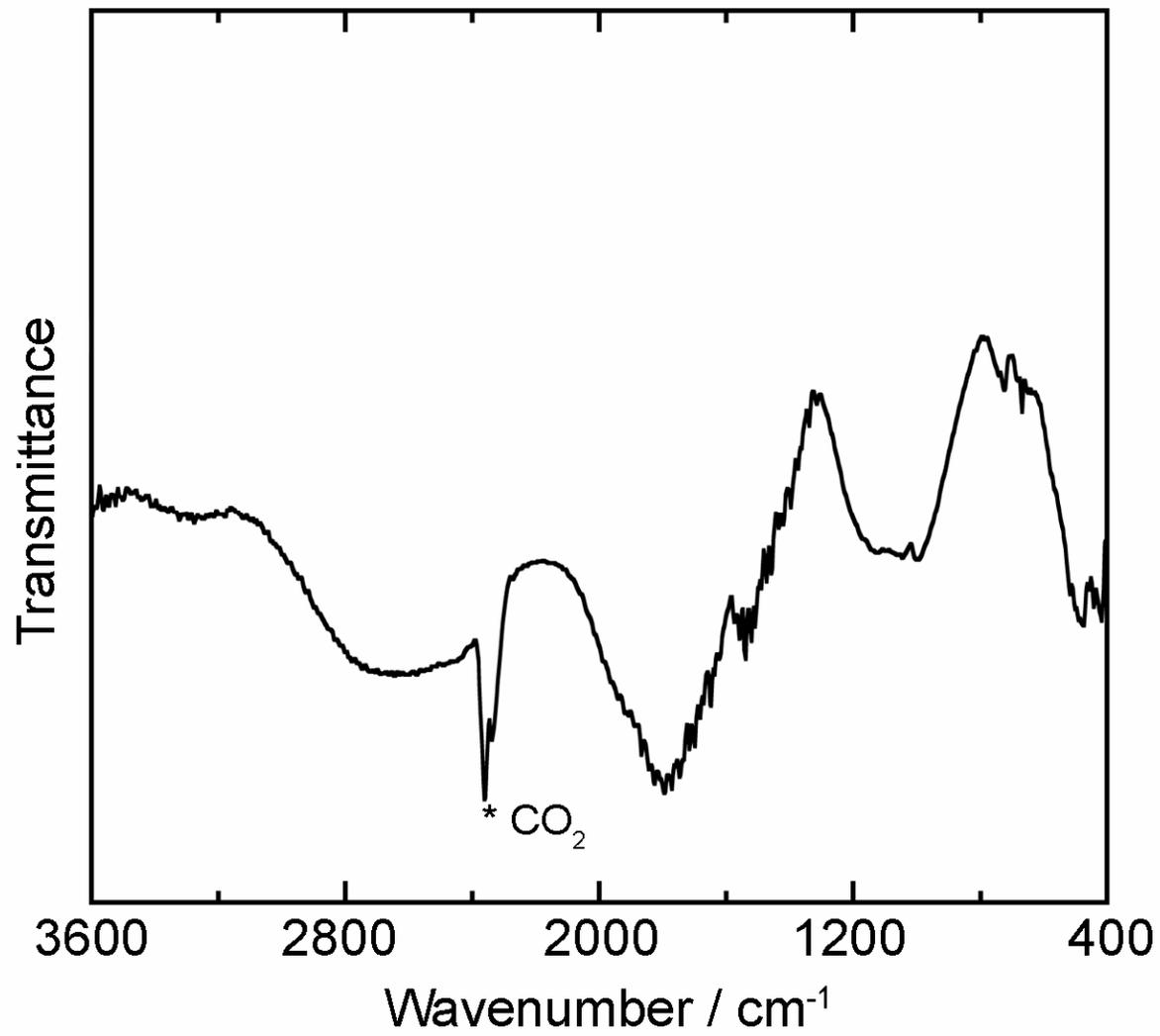


Fig. 2
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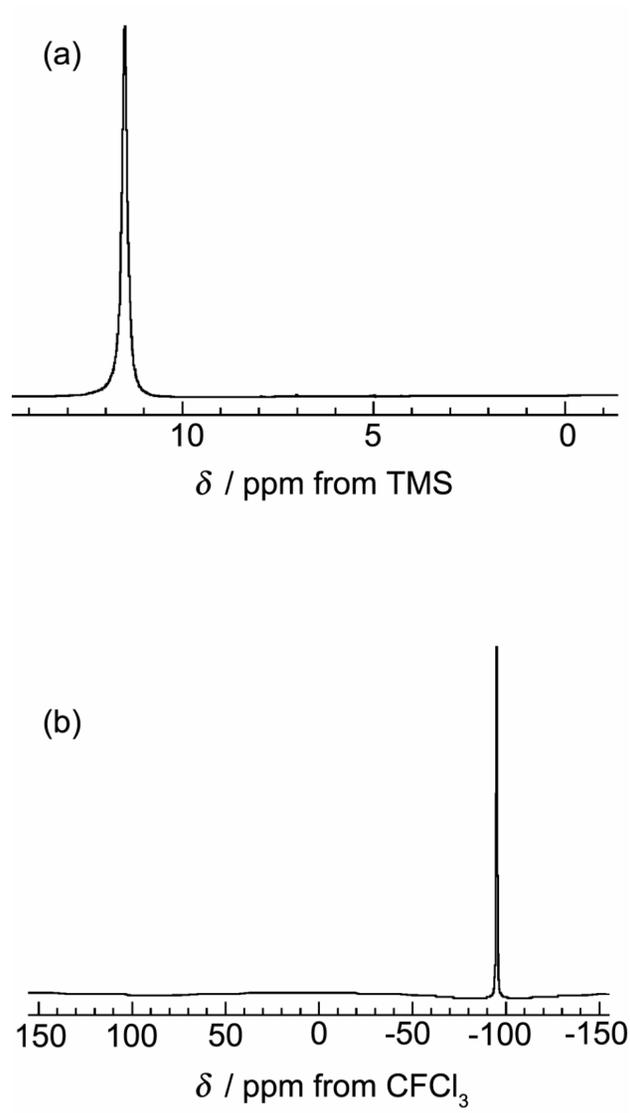


Fig. 3
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Physical and chemical properties are discussed on a room temperature molten fluorohydrogenate, $\text{Cs}(\text{FH})_{2,3}\text{F}$.

