



# Chloride Salt Purification by Reaction With Thionyl Chloride Vapors to Remove Oxygen, Oxygenated Compounds, and Hydroxides

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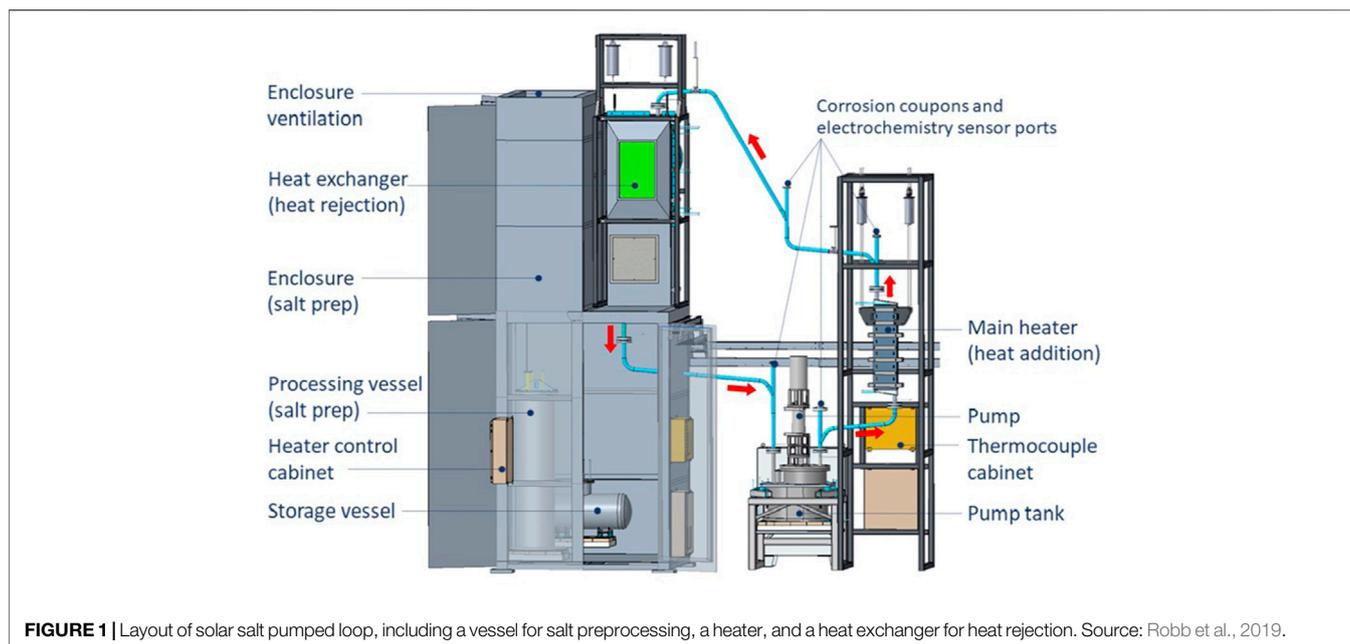
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Molten chloride salts (including  $\text{MgCl}_2$ ,  $\text{KCl}$ ,  $\text{NaCl}$ , and  $\text{ZnCl}_2$ ) are being considered for heat transfer media for renewable (solar) and nuclear power generators, as fuel carrier for nuclear reactors, and as thermal energy storage media. Impurities such as oxygen, hydroxides, moisture, and sulfur are known to negatively influence the corrosion of materials in contact with the salt (e.g., structural metals). Commercially available chloride salts come with a range of impurities. Before using the chloride salts at high temperature, it is desirable to remove the impurities to increase the performance of the salt and reduce corrosion. In this study, we tested the use of thionyl chloride vaporized into a stream of argon to react with oxygenated impurities in a mixture of  $\text{MgCl}_2$ - $\text{KCl}$ - $\text{NaCl}$ , removing them as  $\text{HCl}$  and  $\text{SO}_2$ . The reagent was bubbled through the salt when both above and below the melting point. The reaction was followed using thermocouple data from the salt and by Fourier transform infrared (FTIR) spectroscopy on the exhaust of the reactor. The reaction kinetics were followed by comparing the peaks from  $\text{SO}_2$  product to  $\text{SOCl}_2$  reagent in the FTIR spectra. The purity of the salt was assessed at the end of the purification process by x-ray diffraction and inductively coupled plasma analysis. Although the process was effective in removing the oxygen content of the mixture, ternary compounds were formed in the process, including  $\text{KNiCl}_3$  and  $\text{KMgCl}_3$ . The nickel in  $\text{KNiCl}_3$  came from the reaction between the salt and the nickel vessel. Thus, these experiments suggest that improvements to the process must be made before using  $\text{SOCl}_2$  vapors for the purification of chloride salts.

**Keywords:** carnallite purification, dehydration with  $\text{SOCl}_2$ , chloride salts for thermal energy storage, concentrating solar power, formation of  $\text{KNiCl}_3$

## INTRODUCTION

Concentrating solar power (CSP) for electricity generation has several advantages over distributed networks of photovoltaic panels (Lovegrove and Stein, 2021). CSP facilities have the economy of scale, and they can take advantage of the existing grid infrastructure and the current paradigm of centralized power generation. However, solar in all its forms relies on direct solar irradiance that fluctuates based on weather as well as the diurnal cycle. For concentrating solar to be considered as a

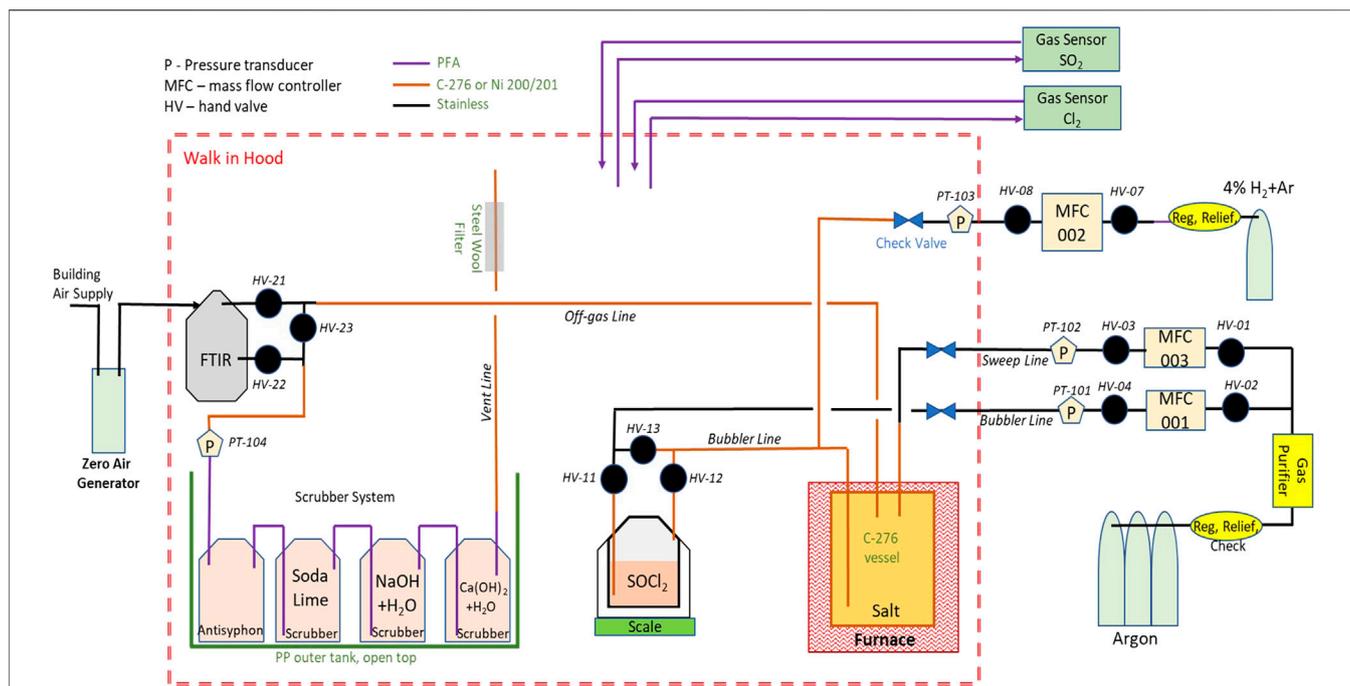


feasible replacement for base-load fossil generation, energy storage must be part of the implementation, either using batteries or thermal energy storage. Molten salt mixtures have been developed for thermal energy storage because of their stability and high operating temperatures, which allow better thermodynamic efficiency in converting heat to electricity.

Sandia National Laboratories (SNL) has been leading research into the use of salts as thermal storage media and has published extensively on this topic (Armijo et al., 2020). The current state-of-the-art for thermal energy storage is a  $\text{NaNO}_3$ - $\text{KNO}_3$  eutectic that can operate up to  $600^\circ\text{C}$ . A range of novel thermal energy storage media have been proposed to improve economics through higher efficiency power conversion (Mehos et al., 2017). Chloride salts are a promising media which allows operation at higher temperatures,  $750^\circ\text{C}$ , and is stable up to  $1400^\circ\text{C}$ . A ternary chloride salt has been studied for this purpose, carnallite, is a mixture of  $\text{NaCl}$ - $\text{MgCl}_2$ - $\text{KCl}$  with a melting point of  $380$ – $426^\circ\text{C}$ . It has the additional advantages of being available at a low cost. However, chloride salts are highly corrosive, especially if there is any oxygen present in the mixture. They also have relatively low thermal conductivity— $0.4\text{ W/mK}$  at  $700^\circ\text{C}$ , which is slightly less than nitrate salts at  $500^\circ\text{C}$ —and an order of magnitude lower than liquid sodium metal. In the SNL analysis of a 2 MWth facility, liquid sodium is passed through a tower receiver placed in the center of a field of solar reflectors. The sodium transfers heat to carnallite or the  $\text{MgCl}_2$ - $\text{KCl}$ - $\text{NaCl}$  mixture. The heated carnallite is either directed towards a super-critical  $\text{CO}_2$  cycle for power generation or to a thermal energy storage reservoir. To support advancement of molten chloride salt technology, Oak Ridge National Laboratory (ORNL) is in the process of building a pumped-chloride test loop, a schematic of which is shown in **Figure 1**. The facility has been filled with  $\text{MgCl}_2$ - $\text{KCl}$ - $\text{NaCl}$  with a nominal composition of (44–50; 48–33; and 2.5–10

weight % respectively), formed by a combination of two commercial salts [anhydrous Carnallite provided by Israel Chemicals Ltd. (ICL) and SPK Halite from Albemarle Inc.]. The nominal salt composition is given in **Supplementary Table S1**.

To reduce corrosion of the metal alloys used to contain chloride salts to acceptable levels, impurities such as oxygen in the form of oxides, hydroxides, and sulfur must be removed to parts-per-million (ppm) concentrations (Kurley et al., 2019; Raiman et al., 2019; Pillai et al., 2021; Ezell et al., 2022). Carnallite cannot simply be dehydrated by heating it above  $100^\circ\text{C}$  because some of the water is chemically bound to the salt components. The options for salt purification were reviewed by Kurley and colleagues (Kurley et al., 2019): for instance,  $\text{MgCl}_2$  is stable as a hexahydrate, the last  $\text{H}_2\text{O}$  of which is very difficult to remove by heating. Various means have been used to strip off the water molecules, including addition of a reducing agent such as magnesium metal or  $\text{Mg}$ - $\text{Cd}$  alloy (Johnson et al., 1969; Zhao et al., 2019). Organic chlorinating agents can also be used, such as reaction with  $\text{CCl}_4$ . The issue with this method is that the byproducts can be toxic organochlorine compounds and the process can become hazardous when operated on an industrial scale. This paper addresses the concern regarding the purity of the chloride salt mixture and investigates a novel way of reducing the oxygen activity in the salt, namely by reacting oxygen in the salt with a sparge of  $\text{SOCl}_2$  vapor in argon. The expected advantages to this method were that the reagent was in the liquid phase and could be handled more easily. Being a liquid allowed it to be introduced by sparging with an inert gas. The  $\text{SOCl}_2$  could be more easily handled in the off-gas of the reactor, with excess reagent being captured downstream of the reactor in a caustic scrubber. The possibility of a lower-temperature reaction, below the melting point of the salt, was also investigated.



**FIGURE 2 |** Schematic of the apparatus used for thionyl chloride purification of chloride salt. Shown in the figure are the vessels holding the salt and the thionyl chloride reagent at the bottom center, gas handling at the right on the outside of the walk-in hood (the dashed red line), post-reaction analysis of the off-gas by FTIR on the left hand side of the figure, and the four-part scrubber gas system (lower left).

## MATERIALS AND METHODS

To better quantify the initial conditions, rather than use the ICL salt, the carnallite to be purified was mixed from individual salts: NaCl (>99.5% Sigma-Aldrich lot #SLBV9985), KCl (>99.5%, Merck lot #K47045236551), and MgCl<sub>2</sub> (>98%, Sigma #SLBX8218). The components were individually loaded into a 15-cm-diameter nickel alloy vessel (C-276) that could be purged with a flow of ultra-high purity (UHP) argon up to 500 cm<sup>3</sup>/min. The sealed vessel was placed into a tubular furnace located in a walk-in fume hood. The complete system is shown in **Figure 2**, and the inside of the hood is delineated as the dashed red line. The system included the heated vessel containing the salt and an unheated glass vessel containing liquid SOCl<sub>2</sub> through which UHP argon carrier gas was bubbled at flow rates up to 400 cm<sup>3</sup>/min. The input gas handling system is shown on the right-hand-side of the figure. The off-gas monitoring and treatment is shown on the left-hand-side of the figure. Because production of acidic gases was anticipated, the argon flow was passed through a series of scrubbers, including a packed soda lime bed, a 2 M NaOH(aq) bubbler, and a 2 M Ca(OH)<sub>2</sub> (aq) bubbler. A steel wool filter was placed in the flow exhausted into the hood to capture any Cl<sub>2</sub> that formed. The hood exhaust also passed through a HEPA filter. A schematic of the overall process is shown in **Supplementary Figure S1**.

The purification experiments took several days; the SOCl<sub>2</sub> was first introduced into the salt when solid. The flow was continued as the salt was heated above its melting point. The various steps are listed in **Table 1**. Two batches of salt were purified: 2.159 g (1,027 g

MgCl<sub>2</sub>, 806 g KCl, and 316 g NaCl) and 6.096 kg (2,875 g MgCl<sub>2</sub>, 2,342 g KCl, and 880 g NaCl). These were chosen to represent the eutectic composition (Mohan et al., 2018). The apparatus was instrumented with three thermocouples measuring the temperature gradient through the furnace, gas flow monitors on the UHP argon, and off-gas monitored by passing it through a transmission gas cell probed by a Bomem FTIR spectrometer. Because the reactions were being monitored frequently, the purification processes were undertaken following several steps over 7 days as indicated in **Table 1**. More than half of that time the SOCl<sub>2</sub> bubbler was by-passed and the reactor was purged with argon. The salt was kept at temperature throughout the run. The SOCl<sub>2</sub> vessel was placed on a balance to measure the total amount used during the runs. Balance readings from the first run indicated that the amount of SOCl<sub>2</sub> that passed through the salt was 134 g; however, that number has a high uncertainty, ± 20%, because the gas line connections may have affected the readings. Thus, the balance was not used in the second run. The amount of SOCl<sub>2</sub> passing through the reactor unreacted was not assessed.

After the purification process, liquid samples were removed from the reactor at 600°C through a sampling tube. There are no standard characterization methods for salt samples, so traditional laboratory methods were used. The samples were sealed under argon and were sent for inductively coupled plasma mass spectrometry (ICP-MS) for determination of elemental composition. The samples were also examined by scanning electron microscopy (SEM) electron dispersive x-ray spectroscopy (EDS), as well as x-ray diffraction analysis (XRD). The SEM-EDS was used to detect oxygen as well as

**TABLE 1** | Test matrix for purification of carnallite.

Temperature range (°C)	Duration	Gas flows
<b>1st purification run (2 kg sample)</b>	<b>7 days total</b>	
Ramp from room temperature to 120°C (bottom) to 150°C (top)	1.5 h for ramp	No $\text{SOCl}_2$ flowing during ramp
Ramp to 190 ± 10°C	14 h at temperature	Salt purge with argon at 375 cm <sup>3</sup> /min
	3 h	
React <sup>1</sup> at 190 ± 10°C	3.75 h	$\text{SOCl}_2$ at 125 cm <sup>3</sup> /min
		Argon at 250 cm <sup>3</sup> /min
Hold at 190 ± 10°C	13 h	Argon at 200–300 cm <sup>3</sup> /min
React at 190 ± 10°C	5.4 h	$\text{SOCl}_2$ at 193 cm <sup>3</sup> /min
		Argon at 180 cm <sup>3</sup> /min
Hold at 190 ± 10°C	1.5 h	Argon at 400 cm <sup>3</sup> /min
React at 190 ± 10°C	5.5 h	$\text{SOCl}_2$ at 100 cm <sup>3</sup> /min
		Argon at 100 cm <sup>3</sup> /min
Hold at 190 ± 10°C	16.75 h	Argon at 400 cm <sup>3</sup> /min
React at 190 ± 10°C	2.25 h—step 10	$\text{SOCl}_2$ at 100 cm <sup>3</sup> /min
		Argon at 100 cm <sup>3</sup> /min
Hold at 190 ± 10°C	2 h	Argon at 200 cm <sup>3</sup> /min
Ramp at 2°C/min to 535 ± 15°C	10 h	Argon at 200–520 cm <sup>3</sup> /min to purge the FTIR
React at 535 ± 15°C	1.5 h	$\text{SOCl}_2$ at 50 cm <sup>3</sup> /min
		Argon at 100 cm <sup>3</sup> /min
Ramp and hold at 640 ± 10°C	3 h	Argon at 100 cm <sup>3</sup> /min
2nd Purification Run (6 kg sample)	7 days total	
Hold at 30°C	3 h	$\text{SOCl}_2$ at 50 cm <sup>3</sup> /min
		Argon at 50 cm <sup>3</sup> /min
Ramp to 180°C and hold	4.6 h	$\text{SOCl}_2$ at 50 cm <sup>3</sup> /min
	11 h	Argon at 150 cm <sup>3</sup> /min
Ramp to 650 ± 10°C	6 h	$\text{SOCl}_2$ at 50 cm <sup>3</sup> /min
		Argon at 150 cm <sup>3</sup> /min
React at 650°C	2 h	$\text{SOCl}_2$ at 40 cm <sup>3</sup> /min
		Argon at 120 cm <sup>3</sup> /min
Hold at 650°C	70 h	Argon at 100 cm <sup>3</sup> /min
React at 500°C	20 h	$\text{SOCl}_2$ at 75 cm <sup>3</sup> /min
		Argon at 150 cm <sup>3</sup> /min

<sup>1</sup>"React" indicates that  $\text{SOCl}_2$  in argon was flowed through the salt vessel. "Hold" denotes an argon purge with no  $\text{SOCl}_2$ .

the other major salt components, and the XRD was used to assess binary or ternary phases. The oxygen content of the purified salt was determined by the standard method of titration with HCl (Kurley et al., 2019). The salts were also assessed after the purification process by thermogravimetric analysis (TGA) using a Setaram instrument to assess whether the water of hydration been removed by the purification process.

## RESULTS

The FTIR proved to be a valuable tool to monitor the reaction as it was progressing; the reagents and the products all had strong infrared signals that were well separated in the mid-IR. The infrared active frequencies used in this study are given in **Table 2**. The transmission of the  $\text{CaCl}_2$  window dropped quickly below 1,000 cm<sup>-1</sup>, but the FTIR still allowed us to distinguish the strong absorptions of  $\text{SO}_2$  and  $\text{SOCl}_2$ .

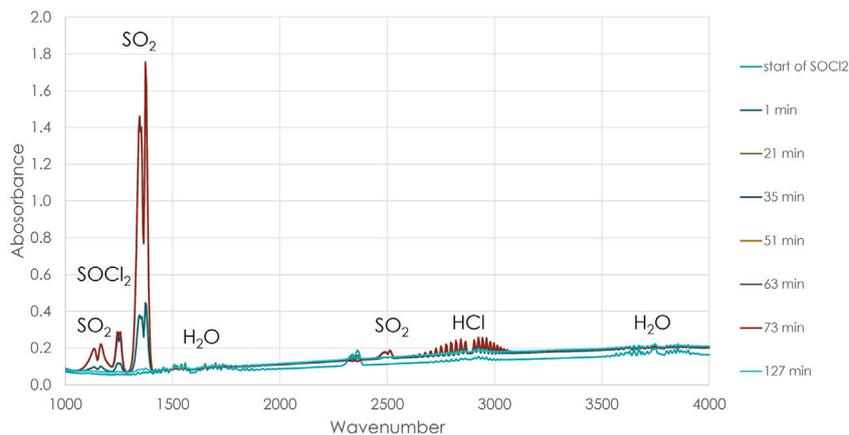
**Figure 3** shows overlaid FTIR spectra from 0 to 127 min after the  $\text{SOCl}_2$  sparge was started through the molten salt. These spectra were normalized to a reference spectrum taken at the start of the experiment before introduction of the  $\text{SOCl}_2$ . The FTIR was purged with argon throughout the experiment, but the increase in  $\text{CO}_2$  over

time shows a slow leak into the body of the instrument. The  $\text{H}_2\text{O}$  peaks are likely derived from free water evolution from the salt, since they did not change much during the experiment. None of the peaks from the products of the purification (i.e.,  $\text{SO}_2$  and HCl) decreased significantly, even after the reaction was terminated. Their signals slowly decreased when the gas cell had been purged with argon for 2 h. This suggested that these gases remained in the gas cell even though it was trace heated to 100°C, perhaps depositing on the  $\text{CaF}_2$  windows.

During the process, the  $\text{SOCl}_2$  signature at 1,250 cm<sup>-1</sup> was quickly replaced with the  $\text{SO}_2$  peaks at 1,150 and 1,300 cm<sup>-1</sup>, showing that there was a rapid reaction of the salt with thionyl chloride. As the reaction progressed, the peaks associated with HCl (2,700–3,100 cm<sup>-1</sup>) also increased. Eventually, the  $\text{SOCl}_2$  was no longer being consumed, and its IR peak started to increase relative to those of the  $\text{SO}_2$ . This indicated that the purification process was complete. When the salt was heated above 600°C, peaks corresponding to HCl and  $\text{H}_2\text{O}$  increased significantly in the FTIR, and a very strong broad IR signal was observed that corresponded to the published spectrum of  $\text{MgCl}_2$  between 3,100 and 3,550 cm<sup>-1</sup>. Unfortunately, the deposition of  $\text{MgCl}_2$  on the gas cell windows meant that the IR spectrometer could no longer be used for the experiment. The increase in the signal of these

**TABLE 2** | Infrared active bands used in this study ( $\text{cm}^{-1}$ ) (Coblentz Society Inc. 2022).

$\text{SOCl}_2$	$\text{SO}_2$	HCl	$\text{H}_2\text{O}$
1251	1120–1160	2610–3070	1250–2070
2480	1300–1400		3400–4000
	2480–2500		

**FIGURE 3** | Overlaid FTIR spectra showing the increased production of  $\text{SO}_2$  over time—the units in the legend are minutes after the  $\text{SOCl}_2$  was valved into the system. Increases were also observed in the HCl peak but were smaller than those for  $\text{SO}_2$ .

species likely arises from reevaporation of deposited material in the tubing coming from the reactor. At the highest reactor temperatures,  $635 \pm 15^\circ\text{C}$ , the top of the crucible was measured as  $507^\circ\text{C}$ . This was high enough to cause secondary volatilization of the reaction products. So, although the FTIR was a useful tool to monitor the progress of the reaction, the deposition and reevaporation of reaction products through the tubing to the gas cell and the long residence time of the reaction products in the gas cell meant that the spectra could not be used for a chemical kinetic analysis.

The purified samples were analyzed by inductively-coupled plasma mass spectrometry (ICP-MS). Results are presented in **Table 3** for the salts before and after the purification processes. Because the ICP-MS could not give oxygen analysis, oxygenated compounds were looked for via SEM and the salt was also analyzed by titration. The SEM shows the presence of oxygen, as shown in **Figure 4**. The analysis of the salt by titration was  $14 \mu\text{g/g}$  in the second test. Note the XRD did not quantitatively capture the presence of sodium chloride because of its lower concentration than the other components.

The salt samples, which were white when introduced into the reaction vessel, became pink after the purification process. The source of the color change was thought to have arisen from an impurity that was absorbed into the melt. The SEM analysis indicated that the metal involved in the color change could be nickel that could have come from the C-276. The XRD analysis shown in **Figure 5** corroborates this analysis, as a potassium-nickel ternary chloride was found in the analysis, or  $\text{KNiCl}_3$ .

A post-purification TGA analysis assessed whether the water of hydration had been stripped from the salt. The results are shown in **Figure 6**. This chart shows that even when handled with

care, the salt quickly absorbs water from the atmosphere, showing up as dehydration peaks between  $100$  and  $200^\circ\text{C}$ . The higher levels of hydration were not observed, which would have been observed above  $300^\circ\text{C}$  had they been present. The sharp peak at  $400^\circ\text{C}$  corresponds to the melting point of the  $\text{KCl-MgCl}_2$  mixture, the main components of the carnallite salt.

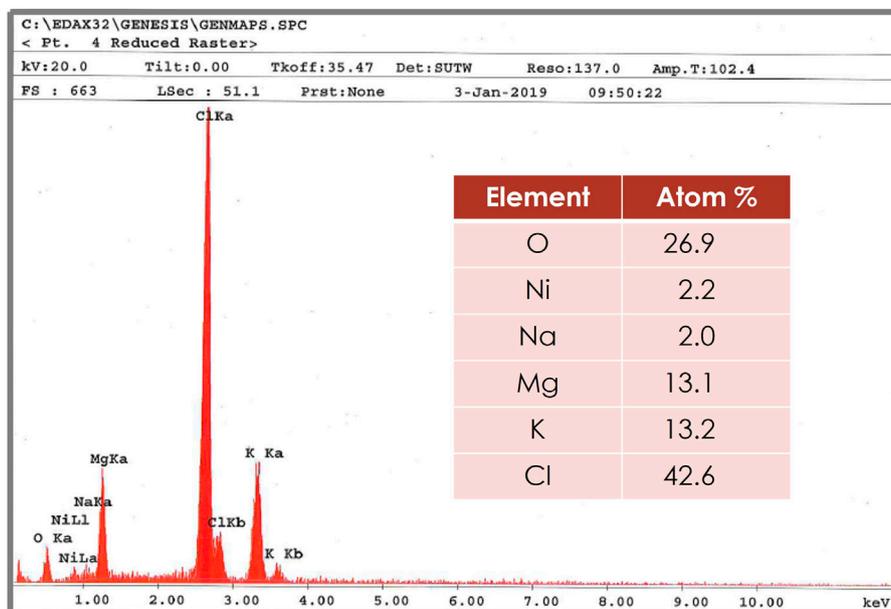
## DISCUSSION

The purification of chloride molten salts needs to be operated under difficult chemical conditions. Kurley, et al. (2019) used thermodynamics to illustrate why HCl alone is not enough to remove  $\text{MgO}$ , the thermodynamically favored product when water is present with  $\text{MgCl}_2$ . The current industrial scale purification of chloride salts is hazardous: using  $\text{Cl}_2$ , HCl, and/or electrical currents to achieve REDOX control, but are themselves hazardous and create hazardous byproducts. The current proposed method in the CSP community using pyrophoric metallic Mg may appear more benign but can generate substantial amounts of HCl off-gas and explosive  $\text{H}_2$ . REDOX control by addition of Mg powder may also result in any unreacted Mg causing abrasion corrosion during flow. Excess Mg may react with containment metals as well as the oxygen-bearing species. Hence, this study investigated the use of a sparged room-temperature liquid,  $\text{SOCl}_2$ , to test a chlorination process to convert hydrates and hydroxides entrained in the salt into vaporized HCl and  $\text{H}_2\text{O}$ .

Sparging the salt with room temperature  $\text{SOCl}_2$  successfully removed hydration  $\text{H}_2\text{O}$  and  $\text{O}_2$  from the salt. The  $\text{O}_2$  content by titration was  $14 \mu\text{g/g}$  after purification, but the salt is extremely hygroscopic and needs protection even during sampling. Most of

**TABLE 3** | Elemental analysis of major and minor components in carnallite salt (wt%-K-Mg-Na).

Element	Synthetic Carnallite (prior to purification)	Purified 1 (2 kg-bulk salt)	Purified 2A (6 kg-bulk salt)	Purified 2B (6 kg-Ni sparging tube)
K	52.2 ± 0.1	42 ± 2	49 ± 3	50 ± 2
Mg	32.4 ± 0.1	42.1 ± 0.4	35.6 ± 0.3	33.7 ± 0.60
Na	15.4 ± 0.1	15.9 ± 0.1	15.1 ± 0.3	16.5 ± 0.2
S	0.001–0.006	0.21 ± 0.01	<Detection limit	<Detection limit
Ca	<Detection limit	$(4.2 \pm 0.02) \times 10^{-2}$	$(1.17 \pm 0.06) \times 10^{-2}$	$(1.10 \pm 0.05) \times 10^{-2}$
Fe	<Detection limit	$(3.45 \pm 0.02) \times 10^{-2}$	$(6.4 \pm 0.01) \times 10^{-2}$	$(6.99 \pm 0.09) \times 10^{-2}$
B	<Detection limit	<Detection limit	<Detection limit	<Detection limit
Cr	<Detection limit	$(5.4 \pm 0.1) \times 10^{-2}$	$(1.61 \pm 0.01) \times 10^{-2}$	$(1.79 \pm 0.09) \times 10^{-2}$
Ni	<Detection limit	54.2 ± 0.3	$(9.01 \pm 0.04) \times 10^{-3}$	0.199 ± 0.001
Ti	<Detection limit	$(2.24 \pm 0.03) \times 10^{-2}$	<Detection limit	<Detection limit
Zn	<Detection limit	$(6.16 \pm 0.03) \times 10^{-1}$	$(2.16 \pm 0.02) \times 10^{-6}$	$(1.64 \pm 0.003) \times 10^{-3}$

**FIGURE 4** | SEM analysis of purified salt showing the main elements including potassium, magnesium, and sodium chlorides. Some oxygen was observed, which was further quantified by titration.

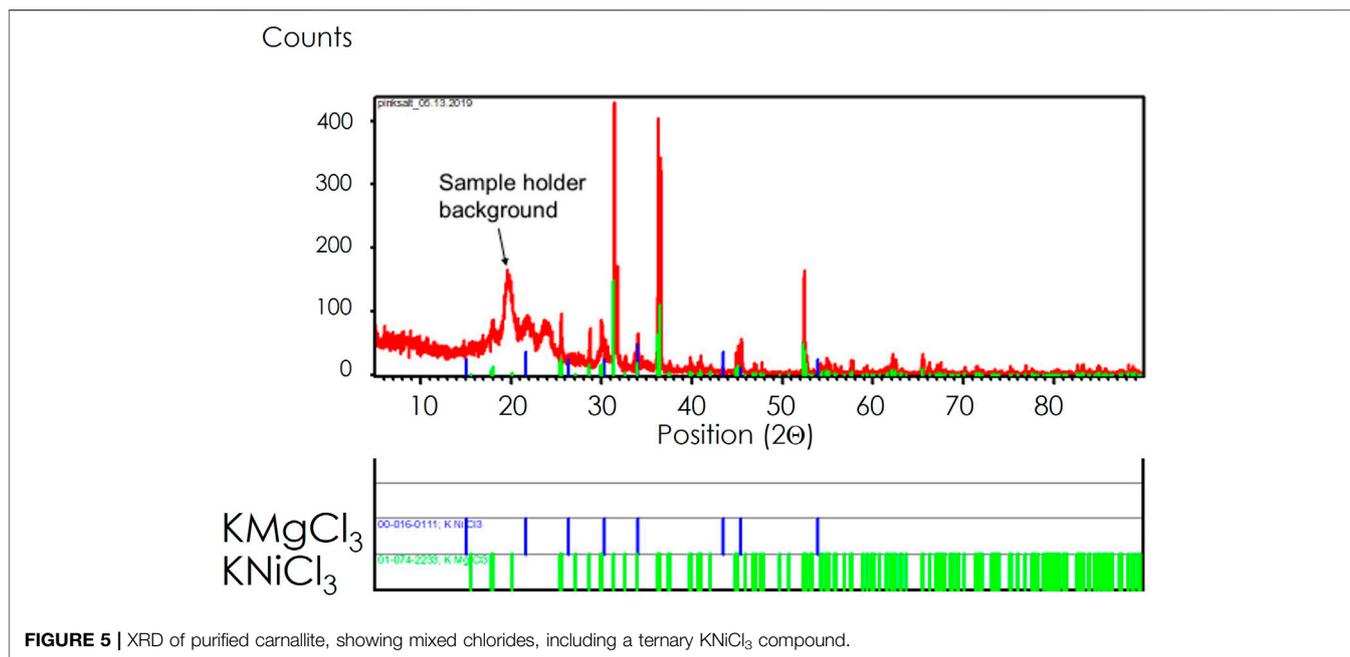
the sulfur from the  $\text{SOCl}_2$  was transported to the scrubber as  $\text{SO}_2$ . Little sulfur was retained in the salt. There appeared to be contamination of the purified salt with nickel coming from reaction of the nickel vessel or sparging tube to form  $\text{KNiCl}_3$ . Because these purification tests were not carried out in different vessels, it is unclear if this reaction was promoted by the presence of  $\text{SOCl}_2$ . Nickel has been reported elsewhere to form Ni(II) chlorides (Seifert, 1988), especially in the presence of acidic gases such as HCl and  $\text{SOCl}_2$  (e.g., Allamagny, 1960) and in mixtures of chloride salts (Lemaire et al., 1997). Removal of the nickel species can be done by filtration, after a secondary  $\text{H}_2$  reduction step if necessary to form an insoluble precipitate.

Results from this study were to have been applied to the purification of the batch of salt used to fill the loop being constructed at ORNL. Although the purification procedure was successful in removing oxygen, it was decided that an alternative measure would be used. The main reasons for this

were the reaction with nickel, a key component of nickel alloy C-276 from which the loop is fabricated, and the difficulties working with  $\text{SOCl}_2$ . Ambrosek (2011) investigated the use of several methods for chloride salt purification, including the addition of magnesium metal to the molten salt. Following this methodology, success has been achieved in the removal of oxides and hydroxides to the Facility to Alleviate Salt Technology Risks (FASTR) molten chloride salt loop at ORNL.

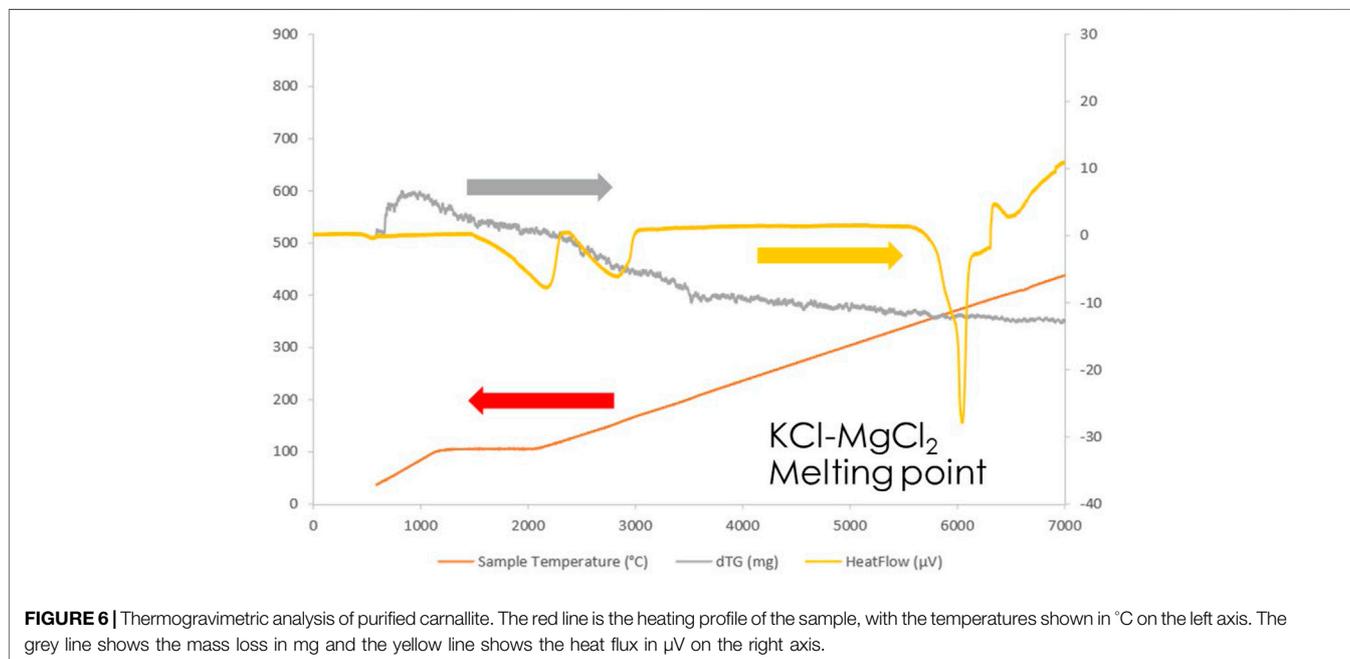
## CONCLUSION

The current approach to removing oxygen-containing species from a mixture of  $\text{MgCl}_2$ -KCl-NaCl for CSP applications is to add copious amounts of Mg, a process that is characterized by large releases of HCl. As the effectiveness of the process will be sensitive to the physical and chemical state of the Mg and the amount of



Mg introduced versus the initial purity of the salt, there will be difficulties in scaling this process to an industrial size. Hence, an alternative purification process was investigated that suggested easier control, as the reagent was a sparged liquid, and better scalability. In this study,  $\text{MgCl}_2$ - $\text{KCl}$ - $\text{NaCl}$  was purified by sparging the molten salt with  $\text{SOCl}_2$  vapor entrained in a stream of argon. The salt purification method was tested with the salt as a solid and as a liquid, about  $150^\circ\text{C}$  above its melting point.  $\text{SOCl}_2$  reacted with the main contribution to bound water, the oxides and hydroxides associated with  $\text{MgCl}_2$ .

Although the oxygen content of the salt was reduced to the ppm level, nickel in the vessel assembly, tubing, and reactor, reacted with the  $\text{SOCl}_2$ , forming a ternary nickel compound,  $\text{KNiCl}_3$ . These effects could be mitigated by choosing different materials for the reactor, and removal of any unwanted precipitates by filtration.  $\text{SOCl}_2$  was easily added to the reaction vessel as a sparged reagent and unreacted vapor was neutralized in the off-gas scrubbing system. However,  $\text{SOCl}_2$  is highly reactive and needs to be stored with care. These effects need to be considered in any scaled-up operation using this purification method.



## DATA AVAILABILITY STATEMENT

The raw data supporting the conclusion of this article will be made available by the authors, without undue reservation.

## AUTHOR CONTRIBUTIONS

JM participated in the experiments, analyzed the data, and drafted the manuscript for publication. GC participated in the experiments. JRM participated in the experiments. RM contributed to the project planning and experimental design. KR participated in the experiments and was principal investigator for the project. DS analyzed the salt

samples after purification. All authors reviewed the manuscript before submission.

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## SUPPLEMENTARY MATERIAL

The Supplementary Material for this article can be found online at: <https://www.frontiersin.org/articles/10.3389/fceng.2022.811513/full#supplementary-material>

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