

***F* and *F*-aggregate color centers in 100 K and room temperature KCl grains irradiated with 10 keV electrons**

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We have conducted experiments on potassium chloride grains under temperature, pressure, and irradiation conditions comparable to planetary and astrophysical environments. These experiments are particularly relevant to the investigation of ocean worlds covered with ice shells that may contain ocean-derived salts and which may be exposed to surface irradiation. Irradiation of sylvite (KCl) under cryogenic conditions yields trapped electrons in *F*- and *M*-center anion vacancies (553 nm and 819 nm, respectively), whereas room temperature measurements after irradiation show additional color-center formation (*R*1 [675 nm], *R*2 [734 nm], and *N* [978 nm]). We attribute these additional features to the merging of *F* centers at higher temperatures, yielding colloids. Potassium chlorate (KClO₃) is identified as a possible radiolytic product resulting from modification of KCl with surficial adsorbed H₂O.

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I. INTRODUCTION

Understanding the formation of color centers in alkali halides is a topic of significant interest in solid state physics [1], and more recently in planetary science and astronomy [2–4]. The hallmark spectroscopic absorptions of color centers in the ultraviolet, visible, and infrared regions provide one of the only diagnostic features for remote detection of otherwise spectroscopically featureless compounds. In addition, salts such as the alkali halides are potentially key indicators of endogenous activity within ice-covered ocean worlds, such as Europa and Enceladus [2,5]. The detection of salts on the surfaces of such worlds is a possible indicator of water-rock interactions that could occur within subsurface oceans and hydrothermally active seafloors [6].

The simplicity of the ionic alkali halide crystal structure leads, in part, to high values for the oscillator strength, *f*, of trapped electrons. For the alkali halides, the trapped electron behavior is analogous to that of a hydrogen atom. The oscillator strength in alkali halides is typically in the range of 0.6 to 0.9, whereas in most minerals exhibiting color due to electronic excitations *f* is of order 10^{−4} [7]. For this reason, low concentrations of trapped electrons in alkali halides can yield strong coloration and distinct absorption features.

Many mechanisms exist for the formation of color centers, including crystal impurities, defects that yield anion or cation vacancies, decay of radiogenic elements that may be minor

species in a mineral, and direct irradiation of the crystal. For the latter two mechanisms, ionizing radiation is obviously needed to yield the free electrons and associated holes. The temperature dependence and type of irradiation are also known to significantly affect the formation and stability of color centers [8].

Formation of the *F* center and its numerous aggregates is well described by the Doorn-Haven model, which holds that *M* centers result from the combination of two *F* centers, and other color centers form through subsequent aggregation of additional *F* centers. The *R* center arises from three *F* centers, and the *N* center from the addition of an *F* center to an *R* center (i.e., four *F* centers). The *F* and *M* centers yield just a single strong absorption band each, while the larger aggregate centers form the *R*1, *R*2, *N*1, and *N*2 bands, respectively, each with a variety of band depths as a function of temperature (*T*), pressure (*P*), irradiation flux, and total dose.

Related to the formation of anion ‘holes’, we also examine the formation of *V* centers, which are the positive (cation) holes, i.e., neutral halogen atom vacancies, that also form during irradiation [9]. We focus on the *V*0 band (also referred to as the *K* band) which is on the shoulder of the *F* band (455 nm).

Here we measure the growth, development, and stability of the above-mentioned bands across the visible to midinfrared region during, and immediately after, irradiation with 10 keV electrons at 100 K, and during warming to room temperature. These results serve to inform our observations of moons of the outer solar system and guide our assessment of the physics and chemistry of their putative subsurface oceans.

A. Methods

Reagent grade sylvite (KCl), with an average grain size of approximately 250 μm was loaded onto a diffuse aluminum substrate at the end of a cryostat coldfinger. The sample and coldfinger were then inserted into a thermovac chamber, the

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†Deceased.

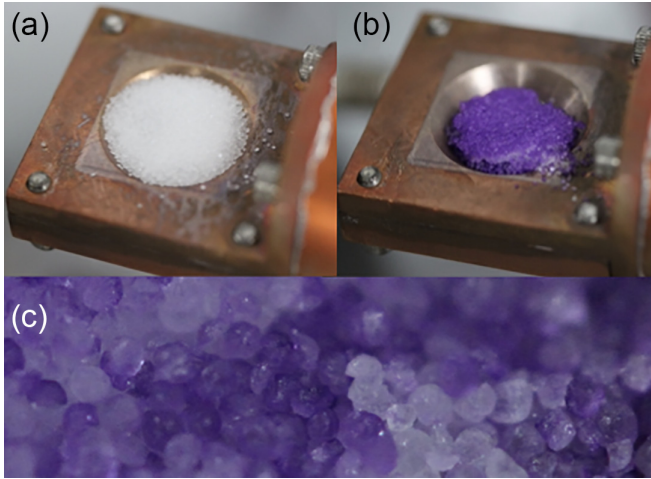


FIG. 1. Sample before and after the irradiation. Prior to irradiation, the sylvite grains were pure white; after irradiation the upper tens of microns of the sample turned deep violet in color. (c) shows a detailed view of the sylvite grains, which are approximately 250 μm in diameter.

details of which are described in Ref. [10] and the Supplemental Material [11].

After sample preparation, the chamber was subsequently reduced to an average internal pressure of 10^{-8} Torr, and the sample was lowered to a temperature of 100 K.

Once at the target temperature and pressure, the sylvite grains were subjected to irradiation by 10 keV electrons, with an average current of approximately 0.5 μA .

The electron beam was rastered over the sample for 18 hours and 15 minutes, yielding a total deposited dose of 2.11 GGy. To alleviate charge buildup on the sample during irradiation, a low-energy (9.9 eV) electron flood gun was directed at the sample throughout irradiation. Before, during, and after irradiation of the sample, visible- to near-infrared spectra, midinfrared spectra, and mass spectra were collected.

B. Results

Figure 1 shows the sample before and after the experiment. Prior to irradiation, the sylvite grains were pure white; after irradiation the upper tens of microns of the sample turned

a deep violet in color. Figure 1(c) shows a detailed view of the sylvite grains after disturbing the sample with a sterile spatula; the surficial grains are violet while the grains below the top layer are still nearly pure white, largely unaffected by the electron irradiation.

Table I summarizes the color centers observed in our experiments at 100 K and 300 K, and provides the wavelength of the band center, as well as comparisons to other published experiments. Supplemental Material Table 1 summarizes published values for the oscillator strength, index of refraction, and the full width at half maximum (FWHM) of the absorption features. These values were used in the calculation of the total concentration of F centers in the irradiated sylvite.

In the sections that follow, we detail each phase of the experiment and the measurements made during those phases.

1. 100 K results and band growth as a function of accumulated dose

To determine the evolution of the color center features during and after irradiation, spectra were examined for changes as a function of accumulated dose and temperature. Figure 2 shows a series of spectra representative of key changes throughout the experiment. All spectra have been normalized to 1022 nm to correct for variations in the white reference background over the course of the experiment. In all spectra, the sample was at 100 K with a chamber pressure of approximately 9.6×10^{-9} Torr.

Prior to irradiation, the sylvite appears nearly flat across the visible and near-infrared region of the spectrum, as shown by spectrum (a) in Fig. 2. This spectrum is the average of seven individual spectra collected before turning on the electron gun.

After seven minutes of irradiation with 10 keV electrons, producing a dose of 13.5 MGy, the sample appeared violet and showed a strong absorption centered at 553 nm. Spectrum (b) in Fig. 2 is an average of 11 spectra taken during this period. Each individual spectrum required fewer than five seconds to acquire. The feature at 553 nm is due to the trapped electrons and indicates the growth of F centers. The corresponding M -center band at 819 nm shows only minor growth, consistent with the model that M centers result from the combination of two F centers [15].

TABLE I. KCl color center formation model and band positions at 100 K and 300 K from this work. Excitation models follow Ref. [12], $v_a^+e^-$ indicates an anion vacancy with a trapped electron; integers then indicate the relative number of vacancies to electron occupancy. Comparison to published values for band positions are shown on the right. n.o. indicates the band was not observed.

Color center	Excitation model	λ_{max} (nm)		Duerig and Markham [13]	Schulman and Compton [1]	Enculescu <i>et al.</i> [14]
		100 K	300 K			
F	$v_a^+e^-$	553	549	540 (28 K)	556 (300 K)	560 (300K)
F' (M)	$v_a^+2e^-$	819	826	820 (300 K)	825 (300 K)	825 (300K)
F_2	$2v_a^+2e^-$	n.o.	n.o.			
F_3 (R_1)	$3v_a^+3e^-$	n.o.	675	680 (300 K)	658 (300 K)	680 (300 K)
F_3 (R_2)	$3v_a^+3e^-$	n.o.	734	730 (300 K)	727 (300 K)	735 (300 K)
F_4 (N_1)	$4v_a^+4e^-$	n.o.	978	980 (80 K)		
F_4 (N_2)	$4v_a^+4e^-$	n.o.	n.o.			
V_0 (K)	$3v_a^+3e^-$	n.o.	455	455 (5 K)	457 (93)	

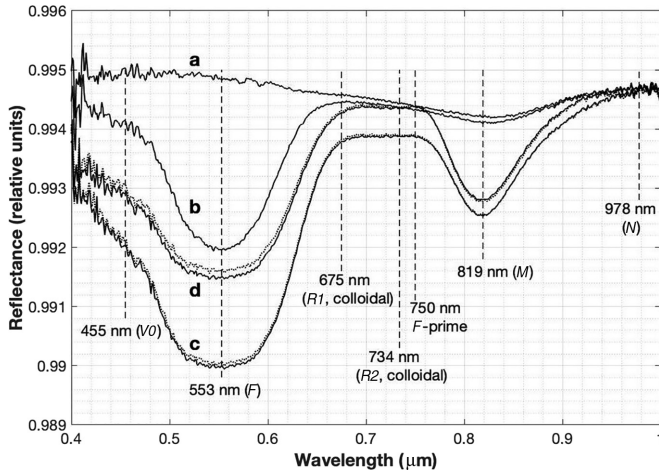


FIG. 2. Formation of the F and M centers, at 553 nm and 819 nm, respectively, during irradiation of KCl with 10 keV electrons. Spectrum (a) shows the KCl grains prior to the onset of irradiation. Spectrum (b) was collected after exposure to a total dose of 13.5 MGy. The two spectra labeled (c) are of the sample after 2.11 GGy, with the upper spectrum in (c) having been collected with the flood gun off (see text). Spectra labeled (d) were collected after the high-energy electron gun was turned off.

The final spectra collected before turning off the electron gun are those labeled (c) in Fig. 2. The lower spectrum, which is an average of six individual spectra, was acquired with the 10 keV beam and the low energy flood gun irradiating the sample. The upper spectrum, which is an average of 11 spectra, was acquired while the 10 keV beam was on, but the flood gun was temporarily off. The slight difference in band depth observed may be attributable to F -center formation from the additional flux of low-energy electrons from the flood gun. We detail this mechanism in Sec. II.

The two spectra labeled (d) in Fig. 2 were collected after the electron gun was turned off. The difference observed between the two d spectra is also attributable to the flood gun and is discussed in Sec. II.

To calculate the number of F centers per cm^3 as a function of accumulated dose, we follow the Smakula-Dexter wave function formulation, using the Dexter model for impurity centers within lattice vibrations of solids [16],

$$N_0 = 0.87 \times 10^{17} \frac{n}{(n^2 + 2)^2} \frac{1}{f_{0j}} \mu_j(\text{max}) U_j, \quad (1)$$

where N_0 is the number of oscillating impurity centers, n is the index of refraction of the host crystal, f_{0j} is the atomic oscillator strength, $\mu_j(\text{max})$ is the peak absorption coefficient, and U_j is the FWHM of the band. In the Dexter form followed here, the absorption band is Gaussian in shape whereas in Smakula's original form a Lorentzian shape was assumed [17].

As shown in Refs. [12,14], the specific lattice parameters for sylvite lead to a generalized form for the number of F centers,

$$N_F(\text{KCl}) = 3.60 \times 10^{15} \times \text{OD}, \quad (2)$$

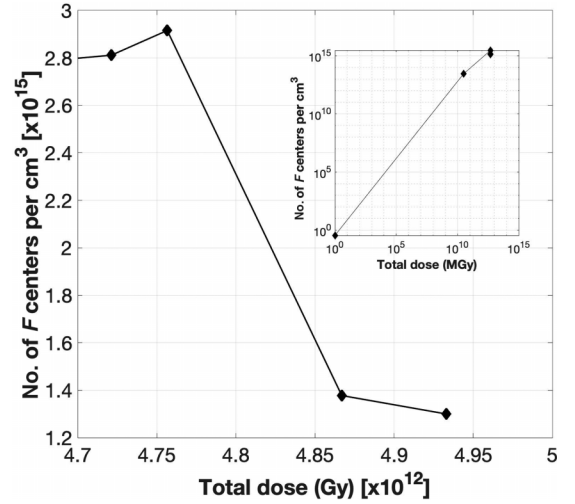


FIG. 3. F center production as a function of total accumulated dose. Inset shows the full experimental range, while the primary plot shows detail on the region in the upper right of the full curve. The small rise in F centers in the primary plot is a result of turning on the 9 eV flood gun (10 keV beam was already on), the subsequent drop in F centers resulted from turning off the 10 keV electron gun while keeping the flood gun on. The final data point corresponds to both the 10 keV beam and the flood gun being turned off.

where OD is the optical depth, which corresponds to the maximum value of the absorbance at the band center.

Reflectance data collected during our experiment was converted to absorbance $[-\ln(R/R_0)]$, and the peak height and full width half maximum were measured, after which a Gaussian deconvolution was conducted to fit the color center peaks. To calculate the total accumulated dose in grey (J kg^{-1}), a total energy deposition thickness of $2 \mu\text{m}$ was used [18], along with a sylvite density of 1.984 g cm^{-3} and a porosity of 0.7.

Figure 3 shows the accumulation, and loss, of F centers as a function of dose. The inset plot shows the rise of F centers over nearly 18 hours of irradiation, followed by a drop in F centers immediately after the shutdown of the electron beam. The full plot shows detail on the cluster of four data points just prior to the end of the 18-hour experiments.

As revealed by these final four data points, the low-energy electrons from the flood gun provided a measurable contribution to both the band depth (as can be seen in Fig. 2) and the total number of F centers (inset in Fig. 3). The first data point in the full plot corresponds to the 10 keV beam being shut off before collection of the corresponding spectra, while the second point (the highest data point in the inset) corresponds to both the 10 keV beam and the 9.9 eV flood gun being on. The next data point (third diamond in the primary plot) was measured after the 10 keV beam was turned off, but the flood gun remained on; the last data point was with both the 10 keV beam and the flood gun turned off.

The sequence of on-and-off for the 10 keV beam and the 9.9 eV flood gun beam provides for a useful quantification of the contribution of low-energy electrons to the total F -center production rate.

The difference between the first two points in the inset sequence is $\Delta N_F = 1.04 \times 10^{14}$. This corresponds to F -center

creation by the 9.9 eV flood gun: the first point was with the 10 keV beam on and flood gun off, the second with both on.

The difference between the last two data points corresponds to a difference in F centers of $\Delta N_F = -7.78 \times 10^{13}$. The decrease here is a result of the 10 keV beam being turned off, while maintaining the flood gun on. In other words, if the high energy beam and flood gun had been turned off at the same time, the plot would have descended directly to the final data point. The final step, i.e., decrease in band depth, is due to the termination of the low-energy electrons no longer producing F centers.

The combination of these two cases clearly shows the role of the low-energy electrons in F center production and maintenance. This is for good reason, based on models of electron penetration depth [18], 9.9 eV electrons should have a mean range of no more than a fraction of an angstrom, making it seemingly impossible for them to penetrate far enough into a crystal lattice to be captured in a vacancy (the potassium atom has a radius of approximately 2.35 Å). Nevertheless, the flood gun electrons clearly contributed to the band depth, and thus the F -center creation and maintenance observed in our experiments.

Interestingly, the additional color center formation is not just a matter of low-energy electrons enabling charge redistribution and deeper penetration of high-energy electrons. Were that the case, then the decrease between the last two data points in Fig. 3 would not have occurred; the high energy gun is not on for either data point, the difference is purely due to the flux of low-energy electrons from the flood gun. We discuss this phenomenon further in Sec. II.

2. 100 K results for band decay after irradiation ceased

The two spectra labeled (d) in Fig. 2 were acquired immediately after the 10 keV electron beam was turned off, with the sample still held at 100 K. The lower spectrum is with the flood gun left on, and the spectrum just above was acquired immediately after the flood gun was shut off. Here again, the difference between the band depths is attributable to the low-energy flood gun electrons.

Both the F - and M -center absorptions decreased rapidly in band depth after the high energy electron gun was turned off [difference between Figs. 2(c) and 2(d)]. The decrease in band depth corresponds to a loss of 1.54×10^{15} F centers. This difference is entirely attributable to the high-energy electron source being turned off—no change in temperature or pressure occurred. For this reason, this difference also serves as a useful metric for the quasi-steady-state color center abundance under persistent irradiation versus that of the steady state when no high-energy electrons are irradiating the sample. The F -center contribution from the 9.9 eV flood gun, as shown in Fig. 3, amounts to an average change of 9.1×10^{13} F centers.

Also significant, no color centers other than F and M were observed during this part of the experiment. This implies that consolidation of F and M centers into larger aggregate color centers was not occurring in either situation: the high-energy electron beam on or off. An important caveat, however, is that the broadening of the absorption, as observed in the spectra of Figs. 2(c) and 2(d), relative to the spectrum in Fig. 2(b) may be attributable to the growth of the $V0$ center at 455 nm. The

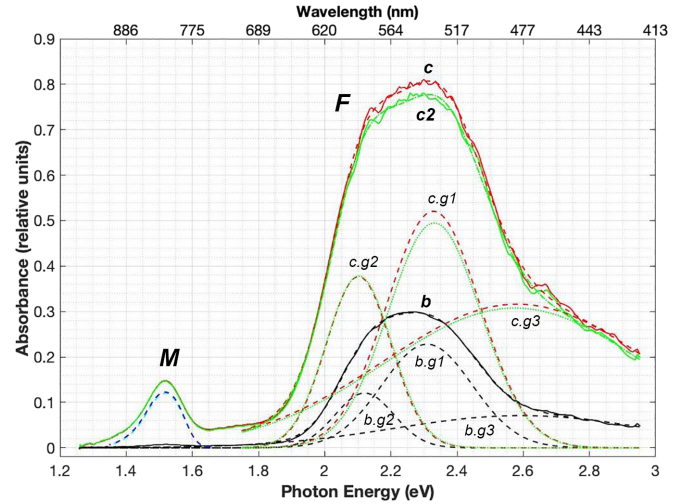


FIG. 4. The shape of the F -center band was best fit by a three-term Gaussian, i.e., three Gaussian curves, $c.g1$, $c.g2$, and $c.g3$, sum to form the fit to the measured curve, c .

predominance of F and M centers at 100 K, and the appearance of the $V0$ center, is consistent with other experiments, which have shown additional color center formation being favorable at higher temperatures (Supplemental Material Table 1).

3. The shape of the F band

The F center band can contain additional bands, one each at the higher and lower energy shoulders of the primary band [1,19].

In our experiments, the F band appeared broad, with a clear extended shoulder at higher energies and a slight broadening at lower energies. The spectra of data from the experiment are shown in absorbance in Fig. 4 with the same letter designations as described in Fig. 2. The solid lines, b , c , and $c2$ (which is grey) correspond to the data. To investigate possible contributions to the pure F band, we modeled the band as a Gaussian curve and found that a three-term Gaussian provided the best fit. The component curves from each fit are each labeled *e.g.*, $c.g1$, $c.g2$, and $c.g3$, indicating the primary, secondary, and tertiary component of the fit to the data of c . For clarity, components for $c2$ are not labeled, but they are represented by the grey dotted curves just beneath each component of c . The dashed lines superimposed over the solid lines of the data are the full summation of the three Gaussian curves that comprise the fit. The $c.g3$ and $b.g3$ curves clearly show the higher energy contribution to the F -center band, while curves $c.g2$ and $b.g2$ indicate a possible contribution from a lower energy band.

Along the shoulder of the F -band spectrum (c in Fig. 4), a small peak was observed at 2.67 eV, and the Gaussian curve corresponding to that region, $c.g3$, has a peak at 2.58 eV. As reported by Schulman and Compton [1], this is likely due to higher excited states of the F -center electrons, which they cite as occurring at 2.71 eV at 97 K. We attribute the peak observed in our experiment to the K -band excitation. The decay of this band, as shown by the difference in curves $c.g3$ and $b.g3$ in Fig. 4, is consistent with this assignment, as

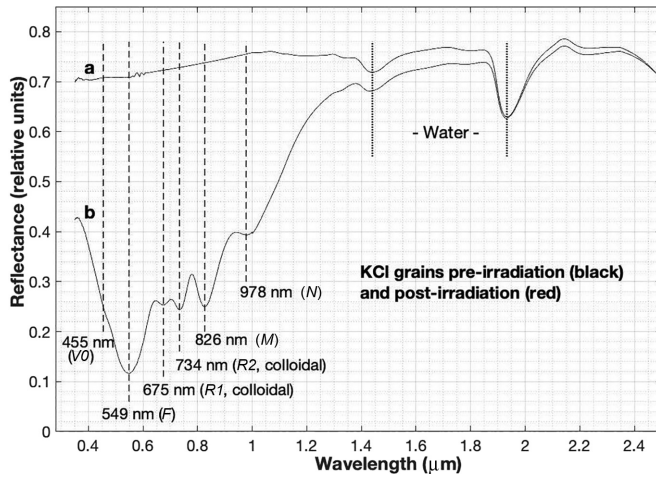


FIG. 5. Room temperature spectra of KCl subsequent to irradiation.

one would expect the excited state to diminish once the high energy electron gun is turned off (curve *b.g.3*).

Band-center positions and FWHM values (shown in brackets) for the *F* center, based on the Gaussian fits, are as follows: $c = 539$ nm (114.6 nm), $c_2 = 549$ nm (120.2 nm), and $b = 548$ (102.5 nm). These values are consistent with those provided in Table I. As discussed above, the shift to higher energy for the band center of c is likely due to contributions from excited state electrons in *F* centers.

With regard to the exact band center position and differences between the published values shown in Table I, we note that we observed a longer wavelength (lower energy) *F* band center at colder temperature relative to the room temperature measurements (553 nm and 549 nm, respectively). This is consistent with our results for the NaCl *F* band position [20], but differs from that of Refs. [13] and [1]. As noted by Ref. [14], with increased radiation flux, the emergence of additional *F* centers and colloidal bands along the shoulders of the primary bands can influence the band center. Future work comparing radiation source, fluence, and flux, all under standardized environmental conditions, is needed to fully understand variations in the band center position.

Lastly, we note that no significant decomposition of the *M* band was observed when modeling the fits represented in Fig. 4. The band center position for the *M* center was found to be at 815 nm, with a FWHM of 60.9 nm.

4. Room temperature results

The visible- to near-infrared spectra collected at room temperature, after the sample was removed from the chamber, are shown in Fig. 5. Spectra of the sample collected before insertion into the chamber are shown for comparison. Broadly, the spectrum of the irradiated sample shows a sharp decline in reflectance shortward of 1500 nm, continuing downward until approximately 500 nm, at which point reflectance sharply rises. This pattern of reflectance explains the distinct violet color of the sample.

The room temperature sample shows numerous absorptions due to the *F* center and associated aggregate color centers. As observed by previous workers [13,15], at higher

temperatures, and upon warming of a sample, the combination of *F* and *M* center electrons yields the aggregate and colloidal color centers, such as the R_1 , R_2 , and *N* centers.

No irradiation occurred during warming from 100 K to 300 K, thus the formation of the aggregate *R* and *N* centers was not due to irradiation or the creation of additional *F* centers. The mechanism for the formation of aggregate color centers as temperature increases beyond 100 K is likely driven by the motion of interstitial ions or atoms during warming, leading to annihilation of some vacancies (e.g., *F* centers) and aggregation of others, creating the R_1 , R_2 , and *N* centers [15]. As detailed in Refs. [14,21], the diffusion of defects during the aggregation process scales with the diffusion coefficient, D :

$$D \sim e^{(-1/T)}.$$

Given the temperature dependence of this process, with warming from 100 K to 300 K, the formation of aggregates increases approximately 0.7% at the higher temperature. While not a large increase, this change in diffusion appears to efficiently yield the observed aggregate color centers. Enculescu *et al.* [14] found that the diffusion of holes is orders of magnitude larger than that of *F* centers, at least for NaCl and LiF. This is consistent with the conclusion of Ref. [15] that aggregate formation is driven by migration of interstitial ions and atoms, as the movement of interstitials is the inverse equivalent of hole migration. In summary, increasing temperature leads to either (1) thermally driven defect and vacancy migration or (2) thermally driven diffusion of *F* and *M* centers to preexisting defects and vacancies in the crystal lattice of KCl.

In our experiment, we observed the following band centers at room temperature (Table I): $F = 549$ nm, $R_1 = 675$ nm, $R_2 = 734.5$ nm, $M = 826$ nm, $N = 978$ nm, and possibly V_0 at 455 nm.

To complement the visible to near-infrared spectra, we also collected midinfrared spectra of the sample throughout the experiment. Figure 6 shows the difference between midinfrared spectra of silvite before and after irradiation. While several possible peaks were observed shortward of 8 μ m, the noise was too large to make useful assignments. Centered at 10.62 μ m (937 cm^{-1}), however, an obvious peak can be seen. We suggest that this peak is likely associated with a product resulting from radiolysis of silvite and trace adsorbed water within the chamber.

Potassium hydroxide (KOH) could be a possible radiolytic product, however, a feature at 11.9 μ m would be expected, which we did not observe, and no known features occur in the 10.6 μ m range [22].

Potassium chlorate, KClO_3 , meanwhile, shows a strong ν_1 feature due to ClO_3^- modes at 10.64 μ m (940 cm^{-1}) at 77 K [23]. We tentatively attribute the 10.62 μ m band in our 100 K experiments to the radiolytic production of potassium chlorate, but more work is needed to confirm this product and to characterize the formation pathway, if confirmed. The potential radiolytic formation of this compound is of significant interest for studies of the surface and subsurface chemistry of icy bodies in the solar system, as discussed below.

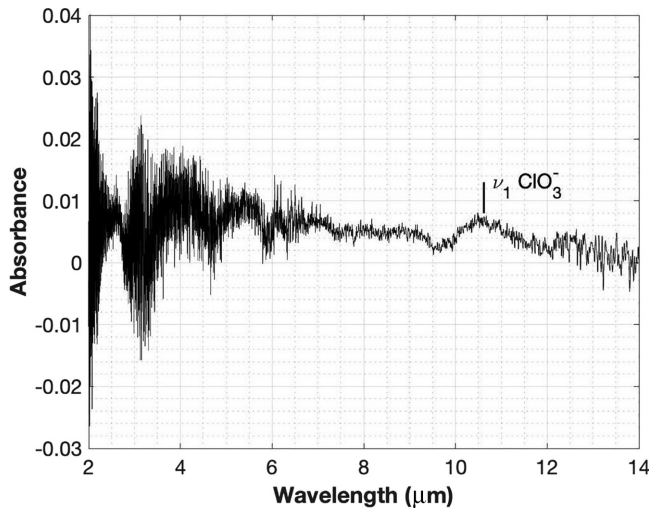


FIG. 6. A difference spectrum, subtracting the preirradiation spectrum from the postirradiation spectrum, reveals a candidate feature at 10.62 μm that could be indicative of radiolytically produced potassium chlorate. The candidate ν_1 feature of the chlorate ion is shown.

II. CONCLUSIONS

We have measured band positions, band depth growth, and total F -center production in sylvite (KCl) as a function of dose at 100 K. The observed F -center band positions of 553 nm at 100 K and 549 nm at 300 K are several nm longer than observed at 28 K (540 nm) [13] and shorter than previously reported at 300 K (556 nm) [1]. F -center aggregates and associated band positions were measured at both 100 K and during warming to room temperature.

Low-energy (9.9 eV) electrons were observed to yield F centers at 100 K, despite low penetration depth into sylvite. As reported by Ref. [24], transport of low-energy electrons in alkali halides can be facilitated by electron-phonon interactions and elastic scattering. The optical (out of phase lattice vibrations) and acoustic (in phase lattice vibrations) phonon interactions were modeled to determine mean free paths (MFPs) as a function of electron energy [24]. For a 9.9 eV electron in KCl, they find the acoustic contribution to the MFP to be 0.35 nm, while the optical contribution is nearly 10 nm.

Though the work detailed in Ref. [24] is for UV photo-produced electrons within the lattice migrating to the surface, we suggest that the same optical-acoustic phonon transport may be responsible for F center production by our 9.9 eV

flood gun in the work presented here [25,26]. The lattice constant of sylvite is 0.629 nm, thus transport of low-energy surface electrons to as much as 10 nm depth within the sylvite lattice could result in electron capture within anion vacancies of the lattice, yielding F centers. Thus, electron transport through electron-phonon interactions may explain the observed small enhancement of F -center production with our flood gun.

The production of potassium chlorate during our experiments is consistent with radiolytic loss of hydrogen from adsorbed water and incorporation of the remaining oxygen into the chlorate anion. Though our experiments did not involve any vapor-deposited water, remnant H_2O at 10^{-8} torr is a likely explanation.

Finally, we note that on ice-covered moons of the outer solar system, several of which likely harbor subsurface oceans, the exposure of endogenous oceanic salts to irradiation on the surface can lead to the production of color centers that are observable remotely with telescopes and spacecraft [2,5,20]. Sylvite color centers have yet to be directly observed, but given the cation abundance of potassium in Earth's ocean [27,28], these strong spectroscopic features are certainly worthy of investigation. The Europa Clipper mission, launched on October 14, 2024, and arriving in the Jovian system in 2030, has as its goal to investigate the habitability of Europa [29,30]. Onboard the spacecraft is an instrument suite with several spectrometers that may be able to detect alkali halide color centers or sample salts ejected from the ice shell [31–33].

Furthermore, potassium chlorate is a strong oxidant, which, if introduced into the ocean of either Europa or Enceladus, could be a useful source of chemical energy for reductant-oxidant reactions that help power biology [34,35].

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DATA AVAILABILITY

All data files and software are available from corresponding author upon reasonable request.

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