

Special Paper

Infrared Spectra and Radiation Stability of H₂O₂ Ices Relevant to Europa

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ABSTRACT

In this paper we present spectra of H₂O₂-containing ices in the near- and mid-infrared (IR) regions. Spectral changes on warming are shown, as is a comparison of near-IR bands of H₂O and H₂O₂-containing ices. An estimate of the *A*-value (absolute intensity) for the largest near-IR feature of H₂O₂ is given. Radiation-decay half-lives are reported for 19 K and 80 K, and are related to the surface radiation doses on Europa. The radiation data show that H₂O₂ destruction is slower at 80 K than 19 K, and are consistent with the claim that icy material in the outermost micrometer of Europa's surface has been heavily processed by radiation. **Key Words:** Europa—Hydrogen peroxide—Radiation effects—Infrared spectra. *Astrobiology* 6, 483–489.

INTRODUCTION

SOLID-PHASE HYDROGEN PEROXIDE (H₂O₂) on Europa (Carlson *et al.*, 1999) is a mixed blessing for astrobiologists. On the one hand, H₂O₂ is made on Europa from H₂O-ice, and so the presence of H₂O₂ strikingly demonstrates a source of potentially biogenic energy at Europa's surface, namely, Jupiter's magnetospheric radiation. In addition, H₂O₂ might undergo both radiolytic and photolytic reactions and thereby serve as a source of molecular oxygen. However, these possibilities are tempered by the fact that H₂O₂ is a powerful oxidant, capable of altering most organic compounds and, essentially, all biomolecules. This suggests that the surface of Europa may be devoid of organic material.

It is not surprising that interest in H₂O₂ among laboratory scientists appears to have increased in

recent years. Aside from its astrobiological possibilities, the H₂O₂ on Europa participates in an intrinsically interesting set of reactions involving H₂O, H₂, O₂, and radicals such as H, OH, and HO₂, which can all be studied in terrestrial laboratories (Johnson *et al.*, 2003). The formation of H₂O₂ from ion-irradiated and ultraviolet-photolyzed H₂O-ices, in both laboratories and on Europa, is thought to proceed either by combination of OH radicals or the O + H₂O → H₂O₂ reaction. However, large energies are not needed to initiate H₂O₂ formation, as shown by Pan *et al.* (2004) in their H₂O₂ synthesis through the action of low-energy electrons (<10 eV) on H₂O-ice.

Reaction products from H₂O₂, such as HO₂, are also of interest because of the aforementioned possibility that H₂O₂ might be a source of O₂. Radiation chemists have long regarded HO₂ disproportionation (HO₂ + HO₂ → H₂O₂ + O₂) as a

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source of O₂ in irradiated liquid H₂O (Swallow, 1973), but Cooper *et al.* (2003) have proposed that in frozen H₂O, dimers of H₂O₂ may be involved. From our own experience (Moore and Hudson, 2003) with the dissociation of isolated CH₃OH and CH₃NH₂, molecules isoelectronic to H₂O₂, the molecular dissociation of H₂O₂ could also contribute to O₂ formation in H₂O-ice.

Although H₂O₂ is certainly relevant to astrobiology concerns for Europa (Cooper *et al.*, 2001), relatively little has been published on the stability and infrared (IR) spectra of this molecule under conditions of astrochemical interest. Older publications include those of Lannon *et al.* (1971) and Giguère and Harvey (1959), which emphasized assignments of H₂O₂ IR bands. Boudin *et al.* (1998) presented mid-IR spectra of two H₂O₂ + H₂O mixtures at a single temperature, but with only estimates for the concentrations. Among the most recent papers, Gomis *et al.* studied H₂O₂ formation with 30 keV ions (2004a) as well as 200 and 400 keV ions (2004b). Loeffler and Baragiola (2005) studied the 3.5- μ m IR band of H₂O₂, while Loeffler *et al.* (2006) reported new experiments on H₂O₂ formation and destruction with 100 keV H⁺.

While all of these earlier papers focused on the mid-IR spectral region (4000–400 cm⁻¹; 2.5–25 μ m), which is useful for product identification, it is the near-IR region (~10000–4000 cm⁻¹; ~1–2.5 μ m) that is more commonly used for remote sensing of icy satellites. Therefore, in this paper we show near- and mid-IR spectra of solid-phase H₂O₂ on the same spectral scale to demonstrate the intensity differences in the two regions. We also estimate the absolute intensity of the strongest near-IR band of H₂O₂, show the strongest near-IR absorbance of H₂O₂ at several temperatures, and compare near-IR bands of H₂O₂ and H₂O in a search for characteristic features. Finally, we present data on the radiation stability of H₂O₂ at two different temperatures. These measurements are intended to provide basic data for future laboratory work and to be of value to both astronomical observers and theorists.

MATERIALS AND METHODS

Briefly, our experiments consist of making thin films of icy materials and examining their IR spectra before and after various radiation and thermal treatments. The procedures used have al-

ready been published (Hudson and Moore, 1995; Hudson *et al.*, 2001, and references therein) so only a summary is given here.

Ice films were formed in three different ways on an aluminum mirror (area ~5 cm²) attached to the tail section of a closed-cycle cryostat in a high-vacuum chamber. To prepare samples of pure H₂O-ice, liquid water was allowed to vaporize in a vacuum system and flow onto the pre-cooled (~10 K) mirror. To prepare pure H₂O₂ ice, a small flask containing the solid urea-H₂O₂ adduct (NH₂)₂CO·H₂O₂ was attached to the high-vacuum chamber. The slow room-temperature decomposition of the compound provided sufficient vapor pressure so that pure H₂O₂ could be deposited onto the 10 K mirror in about an hour (Ball and Massey, 1995). Finally, H₂O + H₂O₂ ice mixtures were made by using a syringe to inject a solution through a vacuum septum onto the pre-cooled aluminum substrate. In all experiments, ice temperatures could be maintained between ~10 and 300 K.

IR spectra of samples were measured by passing an IR beam through the ice film, reflecting it from the underlying aluminum surface, and again passing it through the ice. Bruker (Billerica, MA) Vector 22 and Mattson (Madison, WI) Polaris spectrometers were used, typically at a resolution of 2 or 4 cm⁻¹ and with 60–200 scan accumulations.

Protons (0.8 MeV) from a Van de Graaff accelerator were used to irradiate ices at 19 K and 80 K, as described by Hudson and Moore (1995). Radiation stopping powers (*S*) and ranges adopted in our work were calculated with the SRIM program of Ziegler *et al.* (1985) (see also: <http://www.srim.org/>). Doses, in Mrad units, were determined by measuring the proton fluence (number of p⁺/cm²) at the aluminum substrate and using the following conversion:

$$\begin{aligned} &(\text{number of p}^+/\text{cm}^2) \times (S, \text{MeV cm}^2/\text{g}) \\ &\times (1.602 \times 10^{-14} \text{ Mrad g/MeV}) \end{aligned}$$

Note that the bombarding protons had a much greater range than the sample thicknesses, and so they penetrated the ices and came to rest in the underlying aluminum substrate, not in the ices themselves. See Moore *et al.* (2001) for details on the use of proton radiolysis to mimic cosmic ray bombardment.

Ice thicknesses employed were simply those required to give strong, undistorted IR bands. Pre-

cise, absolute values for the thicknesses were neither measured nor, in general, needed to reach the goals already outlined. Nevertheless, we estimated our samples' thicknesses by simple comparisons (scalings) against IR absorptions of known absolute intensity, such as features of H₂O-ice. These rough estimates showed that our ices ranged from about 0.5 μm to about 5 μm in thickness. More precise measurements, using laser interference fringes, are planned for the future, as are corrections for the differences in reflection and transmission spectra (Loeffler *et al.*, 2006).

The reagents used in our work were as follows: triply distilled H₂O with a resistance greater than 10^7 ohm cm; 30% H₂O₂ (J.T. Baker, Phillipsburg, NJ); 3% H₂O₂, a standard pharmaceutical reagent; and urea-H₂O₂ complex (Aldrich Chemical Co., Milwaukee, WI). The H₂O₂ solution that was nominally 3% was titrated with KMnO₄, and found to be 2.66% by mass or 1.4% by number, relative to H₂O. For convenience, we will refer to this dilute H₂O₂ mixture simply as 3% H₂O₂.

The relative uncertainty in concentrations of our H₂O₂ solutions, measured by titration, was well under 1%. Other uncertainties and sources of error concern IR bands and radiation doses. Positions of sharp bands could be measured to within a few wavenumbers, but positions of broad, weak near-IR features are probably good to only ± 5 cm^{-1} . The relative error for band integrations is around 1%. Temperature measurements are accurate to about ± 1 K. Incident radi-

ation doses, in p^+/cm^2 , are accurate to within a few percent, but their conversion to Mrad units is more problematic. This conversion relies (see above) on an accurate stopping power, S in $\text{MeV cm}^2/\text{g}$, which may not be more accurate than 5%. In the end we adopted a safe, albeit pessimistic, value of 10% for the uncertainty in our stopping power. [See Ziegler *et al.* (1985) and Mozumder (1999) on uncertainties of stopping power for compounds and mixtures.]

RESULTS

Figure 1 shows IR spectra of two pure amorphous H₂O₂ ices, corresponding to two different sample thicknesses (~ 0.5 μm and ~ 1 μm). Mid-IR absorbances are at 3262, 2821, 1361, 886, and 600 cm^{-1} , in agreement with previous low-temperature spectra (Giguère and Harvey, 1959; Lannon *et al.*, 1971). A weak near-IR feature at 4725 cm^{-1} can be seen in the upper trace of Fig. 1, which is reasonably close to the 4775 cm^{-1} value reported for room-temperature liquid H₂O₂ (Bain and Giguère, 1955). Comparing the area of the narrow 2821 cm^{-1} band with that of the broad 4725 cm^{-1} band showed them to be in a ratio of about 6.5:1. Figure 2 shows that the near-IR band at 4725 cm^{-1} weakens slightly and shifts on warming the sample from 9 K to 200 K. A distinct change takes place above 125 K, which is consistent with a 140 K amorphous-to-crystalline phase transformation for solid H₂O₂ (Ghormley, 1957).

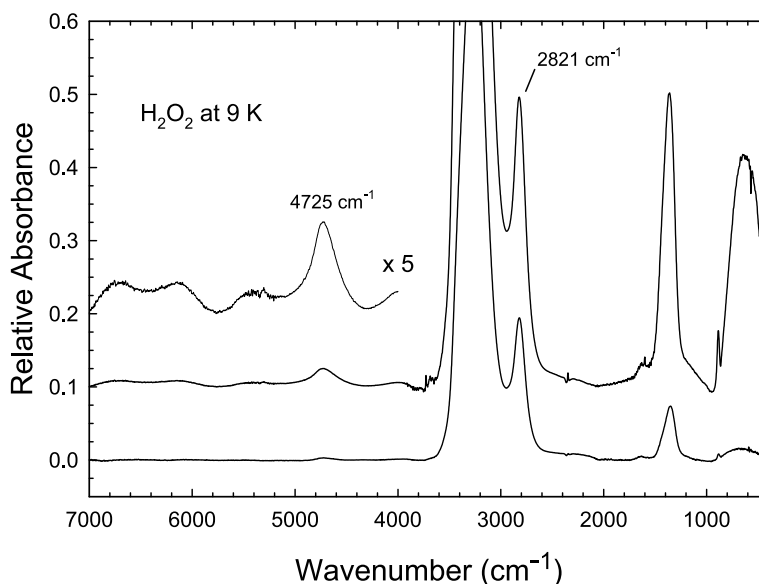
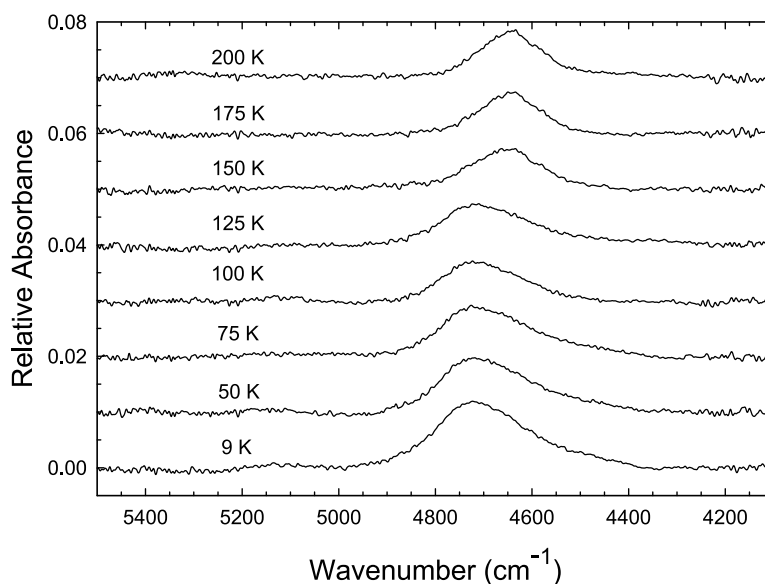


FIG. 1. Spectra of thin (~ 0.5 μm , bottom spectrum) and thick (~ 1 μm , top spectrum) H₂O₂ samples. A few small artifacts, mostly from atmospheric contaminants, are in the upper spectrum near 5350, 3700, 2348, 1601, and 570 cm^{-1} . Also shown is a fivefold enlargement of the near-IR region of the thick sample's spectrum.

FIG. 2. Warming sequence for a pure H_2O_2 ice.



Warming pure H_2O_2 to 223 K resulted in complete sublimation of the sample in about 10 min.

Figure 3 shows near-IR features of ices with three different compositions: 100% H_2O_2 , 30% H_2O_2 , and pure H_2O (0% H_2O_2). These spectra are presented with a greater vertical expansion than those of Fig. 2 and with a much larger wavenumber range. The very broad, almost coincident bands for pure H_2O_2 and pure H_2O are similar to what has been seen in the liquid phase for these two compounds (Bain and Giguère, 1955). Near-IR spectra of 3% H_2O_2 (not shown) proved to be essentially the same as those for pure H_2O .

In an earlier paper (Moore and Hudson, 2000), we investigated the formation of H_2O_2 in irradiated H_2O , but the radiation-induced *destruction* of H_2O_2 is also of interest. Figure 4 shows a first-order decay plot of H_2O_2 destruction in a 3% H_2O_2 - H_2O ice at 19 K and 80 K. For these experiments, the decrease in the H_2O_2 IR band at ~ 2850 cm^{-1} was followed.

DISCUSSION

One goal of this work was to estimate the absolute intensity of the strongest near-IR feature of

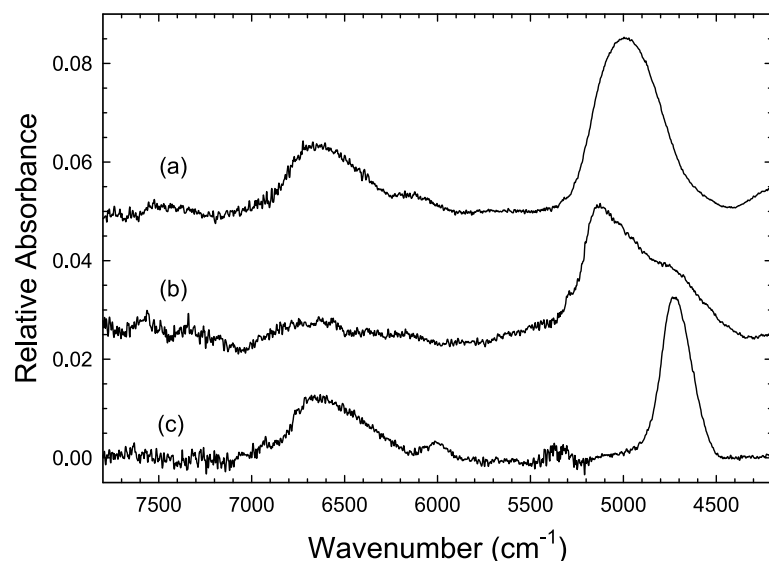


FIG. 3. Near-IR spectra at ~ 10 K for samples with three different H_2O_2 concentrations: (spectrum a) pure H_2O , (spectrum b) 30% H_2O_2 , and (spectrum c) 100% H_2O_2 .

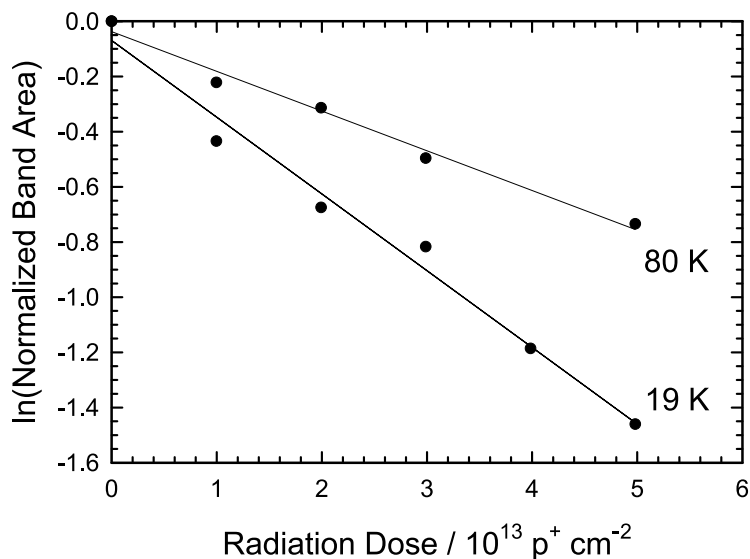


FIG. 4. Radiation-induced decay in 3% H₂O₂ by 0.8 MeV H⁺.

H₂O₂. An absolute intensity, usually denoted A with units of cm/molecule, is useful as it allows quantification of an observed IR feature through the following equation:

$$A = \frac{\int \tau(\tilde{\nu}) d\tilde{\nu}}{N}$$

Here N is the column density of the sample, in molecules/cm², and $\int \tau(\tilde{\nu}) d\tilde{\nu}$ is found by measuring the area of an IR band (in cm⁻¹) and multiplying by 2.303. [See Grim *et al.* (1989) for additional details.] In an earlier paper, we measured $A(\text{H}_2\text{O}_2, 2850 \text{ cm}^{-1}) = 2.7 \times 10^{-17} \text{ cm/molecule}$ for a 3% H₂O₂ + H₂O mixture (Moore and Hudson, 2000). Using this A value, we find $A(\text{H}_2\text{O}_2, 4725 \text{ cm}^{-1}) = (2.7 \times 10^{-17} \text{ cm/molecule})/6.5 = 4.2 \times 10^{-18} \text{ cm/molecule}$, since the 2821 and 4725 cm⁻¹ bands are in a ratio of about 6.5:1. This near-IR A value is only an estimate due to differences in sample composition, but since all H₂O and H₂O₂ ices should have similar structures dominated by hydrogen bonding, the actual A value is not likely to differ by much. Although this estimate is for spectra taken near 10 K, the 4725 cm⁻¹ band's position and profile scarcely change up to ~140 K (Fig. 2). We also note that our $A(\text{H}_2\text{O}_2, 10 \text{ K}, 4725 \text{ cm}^{-1}) = 4.2 \times 10^{-18} \text{ cm/molecule}$ is the same order of magnitude as $A(\text{H}_2\text{O}, 10 \text{ K}, 5040 \text{ cm}^{-1}) = 1.2 \times 10^{-18} \text{ cm/molecule}$ recently reported for an overtone of H₂O (Gerakines *et al.*, 2005).

Recently, Loeffler *et al.* (2006) measured $A(\text{H}_2\text{O}_2, 80 \text{ K}, \sim 2850 \text{ cm}^{-1}) = 5.2 \times 10^{-17} \text{ cm/molecule}$, which is about twice the value we

reported earlier (Moore and Hudson, 2000). Using this newer value for our work here, we obtain $A(\text{H}_2\text{O}_2, 4725 \text{ cm}^{-1}) = (5.2 \times 10^{-17} \text{ cm/molecule})/6.5 = 8.0 \times 10^{-18} \text{ cm/molecule}$. The techniques and H₂O₂ sources for obtaining these two $A(\text{H}_2\text{O}_2)$ values were different, so it is hoped that they can be compared more closely through further additional work in our laboratory and the publication of additional details "in a future paper" mentioned by Loeffler *et al.* (2006).

Another goal of the present paper is to seek clear differences between the near-IR absorbances of H₂O₂ and H₂O. The strongest bands for pure H₂O₂ (4725 cm⁻¹) and pure H₂O (5000 cm⁻¹) in Fig. 3 do show differences in position and profile. For the small H₂O₂ abundances that occur on Europa and are expected to occur on Mars and in the outer Solar System, however, the near-IR spectra of H₂O₂ + H₂O mixtures are essentially the same as those of pure H₂O. We conclude that the prospects for using the 7500–4500 cm⁻¹ region to study H₂O₂ in H₂O-dominated ices are poor.

In our earlier work we showed that the equilibrium H₂O₂ abundance reached in 800 keV H⁺-irradiated H₂O-ice was greater at 16 K than at 80 K (Moore and Hudson, 2000). A similar result has since been reported with 100 keV H⁺ (Loeffler *et al.*, 2006) and 200 keV H⁺ (Gomis *et al.*, 2004b). However, it has also been found (Gomis *et al.*, 2004a) that the H₂O₂ abundance trend seen with 30 keV H⁺ is not the same as for the higher-energy ions. We agree with the suggestion (Gomis *et al.*, 2004a; Loeffler *et al.*, 2006) that each inci-

TABLE 1. RADIATION-INDUCED DECAY OF H₂O₂ IN H₂O-ICE

T (K)	Slope in Fig. 4 (cm ² /p ⁺)	t _{1/2} (p ⁺ /cm ²)	t _{1/2} (Mrad) ^a
80	-1.44 × 10 ⁻¹⁴	4.81 × 10 ¹³	211
19	-2.78 × 10 ⁻¹⁴	2.49 × 10 ¹³	109

Conversions were as follows: 1.0 × 10¹³ p⁺ cm² incident = 43.9 Mrad = 0.439 MGy = 0.082 eV/H₂O molecule = 0.092 eV/16-amu molecule.

^aA relative uncertainty of ~10% should be attached to these values because of uncertainty in the stopping power adopted. See Materials and Methods.

dent ion's stopping power, and the role of elastic collisions, may be the source of the variation. In our earlier H₂O₂ paper (Moore and Hudson, 2000), we also described how scavenging experiments suggest that electron-transfer reactions are important for explaining the differences in H₂O₂ abundance at 16 K and 80 K. Loeffler *et al.* (2006) favor an explanation based on reactions of neutral free radicals, but we know of no scavenging experiments to help decide the issue.

In our previous paper (Moore and Hudson, 2000), we also suggested that the lower H₂O₂ abundance at the higher temperature might be due to a greater rate of H₂O₂ destruction. In the present work, H₂O₂ destruction was studied at two temperatures, and the results are summarized in Fig. 4. To convert the *x*-axis units to more conventional measures of absorbed radiation dose, we took the stopping power (energy loss) of 0.8 MeV protons in solid 3% H₂O₂ ice to be the same as in pure H₂O-ice. This value was calculated to be 274 MeV cm²/g using the method of Ziegler *et al.* (1985). This stopping power led to the following conversions between incident radiation fluence and absorbed dose in Mrad, MGy, and eV units: 1.0 × 10¹³ p⁺ cm² incident = 43.9 Mrad = 0.439 MGy = 0.082 eV/H₂O molecule = 0.073 eV/16-amu molecule.

The slope of each line in Fig. 4 is the negative of the decay constant at the temperature indicated, and the radiation half-life is t_{1/2} = -ln(2)/slope in each case. Table 1 gives slopes and half-lives at the two temperatures used in these experiments. Our earlier suggestion (Moore and Hudson, 2000) that H₂O₂ destruction might be faster at the higher temperature is contradicted by the data summarized in Fig. 4 and Table 1. The smaller H₂O₂ abundance seen (Moore and Hudson, 2000) after H₂O-ice is irradiated at 80 K, as compared with ~19 K, is apparently due to a less efficient formation pathway as opposed to a

greater rate of destruction, though the mechanistic details remain unknown.

Table 2 converts our laboratory t_{1/2} value for radiation-induced H₂O₂ destruction to the values expected at different depths below the surface of Europa, based on the work of Cooper *et al.* (2001). It is apparent from Table 2 that H₂O₂ at a depth of 1 cm on Europa would be largely destroyed in a few years in the absence of reactions involving other molecules (*i.e.*, H₂O). Since H₂O₂ is observed at the present, then its efficient formation is suggested, aided either by the presence of other molecules within the irradiated H₂O-ice (Moore and Hudson, 2000) or by jovian magnetospheric ions having greater stopping power than the H⁺ used in this study (Gomis *et al.*, 2004a,b; Loeffler *et al.*, 2006). Additional work is needed to test these possibilities. In any case, since the thicknesses of our ice samples are similar to the depth of Europa probed by mid-IR observations, the radiation-induced changes we detected are comparable to those in the ices observed by remote IR sensing of Europa.

To summarize, we have presented new low-temperature spectra of H₂O₂ + H₂O-ices show-

TABLE 2. HALF-LIVES FOR H₂O₂ ON EUROPA

Depth below surface (cm) ^a	Dose rate (Mrad/month) ^b	t _{1/2} (days) ^c
10 ⁻⁴	600	0.35
10 ⁻³	200	1.06
10 ⁻²	40	5.28
10 ⁻¹	3	70.3
10 ⁰	0.3	703

^aAssumes an ice density of 1 g/cm³.

^bFrom Fig. 16 of Cooper *et al.* (2001).

^cUsing t_{1/2} (80 K) = 211 Mrad in Table 1 of the present paper. A relative uncertainty of ~10% should be attached to these values because of uncertainty in the stopping power adopted. See Materials and Methods.

ing variations due to concentration and temperature. A near-IR band strength estimate has been made, along with measurements of radiation half-lives at two temperatures. Our work is consistent with the conclusion of others (e.g., Carlson *et al.*, 1999; Cooper *et al.*, 2001) that icy material in the outermost micrometer of Europa has been heavily processed by radiation.

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