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Infrared Spectra and Radiation Stability of H_2O_2 Ices Relevant to Europa

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ABSTRACT

In this paper we present spectra of H_2O_2-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 H_2O and H_2O_2-containing ices. An estimate of the *A*-value (absolute intensity) for the largest near-IR feature of H_2O_2 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 H_2O_2 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 (H_2O_2) on Europa (Carlson *et al.*, 1999) is a mixed blessing for astrobiologists. On the one hand, H_2O_2 is made on Europa from H_2O-ice, and so the presence of H_2O_2 strikingly demonstrates a source of potentially biogenic energy at Europa's surface, namely, Jupiter's magnetospheric radiation. In addition, H_2O_2 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 H_2O_2 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 H_2O_2 among laboratory scientists appears to have increased in recent years. Aside from its astrobiological possibilities, the H_2O_2 on Europa participates in an intrinsically interesting set of reactions involving H_2O, H_2, O_2, and radicals such as H, OH, and HO_2, which can all be studied in terrestrial laboratories (Johnson *et al.*, 2003). The formation of H_2O_2 from ion-irradiated and ultraviolet-photolyzed H_2O-ices, in both laboratories and on Europa, is thought to proceed either by combination of OH radicals or the $O + H_2O \rightarrow H_2O_2$ reaction. However, large energies are not needed to initiate H_2O_2 formation, as shown by Pan *et al.* (2004) in their H_2O_2 synthesis through the action of low-energy electrons (<10 eV) on H_2O-ice.

Reaction products from H_2O_2, such as HO_2, are also of interest because of the aforementioned possibility that H_2O_2 might be a source of O_2. Radiation chemists have long regarded HO_2 disproportionation ($HO_2 + HO_2 \rightarrow H_2O_2 + O_2$) as a

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source of O_2 in irradiated liquid H_2O (Swallow, 1973), but Cooper *et al*. (2003) have proposed that in *frozen* H_2O, dimers of H_2O_2 may be involved. From our own experience (Moore and Hudson, 2003) with the dissociation of isolated CH_3OH and CH_3NH_2, molecules isoelectronic to H_2O_2, the molecular dissociation of H_2O_2 could also contribute to O_2 formation in H_2O-ice.

Although H_2O_2 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 H_2O_2 IR bands. Boudin *et al*. (1998) presented mid-IR spectra of two H_2O_2 + H_2O mixtures at a single temperature, but with only estimates for the concentrations. Among the most recent papers, Gomis *et al*. studied H_2O_2 formation with 30 keV ions (2004a) as well as 200 and 400 keV ions (2004b). Loeffler and Baragiola (2005) studied the 3.5-μm IR band of H_2O_2, while Loeffler *et al*. (2006) reported new experiments on H_2O_2 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 μm), which is useful for product identification, it is the near-IR region (~10000–4000 cm^{-1}; ~1–2.5 μm) 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 H_2O_2 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 H_2O_2, show the strongest near-IR absorbance of H_2O_2 at several temperatures, and compare near-IR bands of H_2O_2 and H_2O in a search for characteristic features. Finally, we present data on the radiation stability of H_2O_2 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 already 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 cm^2) attached to the tail section of a closed-cycle cryostat in a high-vacuum chamber. To prepare samples of pure H_2O-ice, liquid water was allowed to vaporize in a vacuum system and flow onto the precooled (~10 K) mirror. To prepare pure H_2O_2 ice, a small flask containing the solid urea-H_2O_2 adduct $(NH_2)_2CO{\cdot}H_2O_2$ was attached to the high-vacuum chamber. The slow room-temperature decomposition of the compound provided sufficient vapor pressure so that pure H_2O_2 could be deposited onto the 10 K mirror in about an hour (Ball and Massey, 1995). Finally, H_2O + H_2O_2 ice mixtures were made by using a syringe to inject a solution through a vacuum septum onto the precooled 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^+/cm^2) at the aluminum substrate and using the following conversion:

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

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. Precise, 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 H_2O-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 H_2O with a resistance greater than 10^7 ohm cm; 30% H_2O_2 (J.T. Baker, Phillipsburg, NJ); 3% H_2O_2, a standard pharmaceutical reagent; and urea-H_2O_2 complex (Aldrich Chemical Co., Milwaukee, WI). The H_2O_2 solution that was nominally 3% was titrated with $KMnO_4$, and found to be 2.66% by mass or 1.4% by number, relative to H_2O. For convenience, we will refer to this dilute H_2O_2 mixture simply as 3% H_2O_2.

The relative uncertainty in concentrations of our H_2O_2 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 $\pm$ 5 cm^{-1}. The relative error for band integrations is around 1%. Temperature measurements are accurate to about $\pm$ 1 K. Incident radiation 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 MeV cm^2/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 H_2O_2 ices, corresponding to two different sample thicknesses ($\sim$0.5 μm and $\sim$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 H_2O_2 (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 H_2O_2 (Ghormley, 1957).

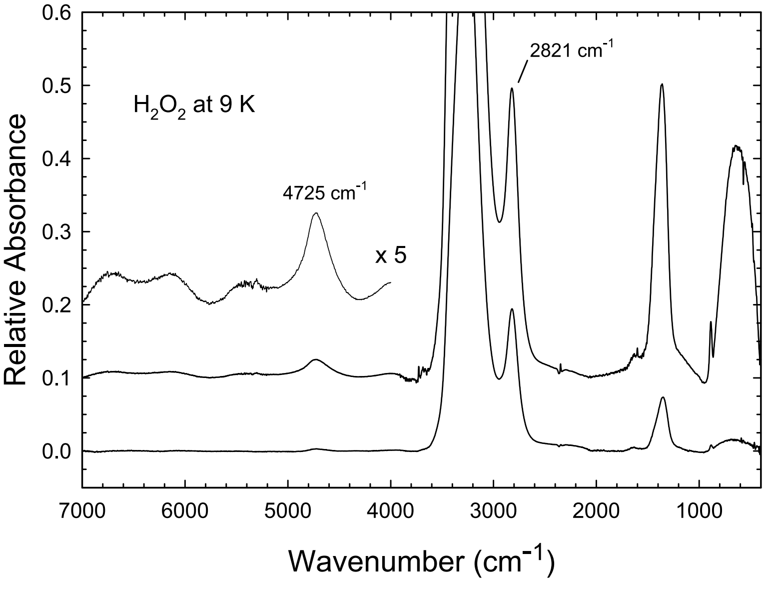

FIG. 1. Spectra of thin ($\sim$0.5 μm, bottom spectrum) and thick ($\sim$1 μm, top spectrum) H_2O_2 samples. A few small artifacts, mostly from atmospheric contaminants, are in the upper spectrum near 5350, 3700, 2348, 1601, and 570 cm^{-1}. Also shown is a fivefold enlargement of the near-IR region of the thick sample's spectrum.

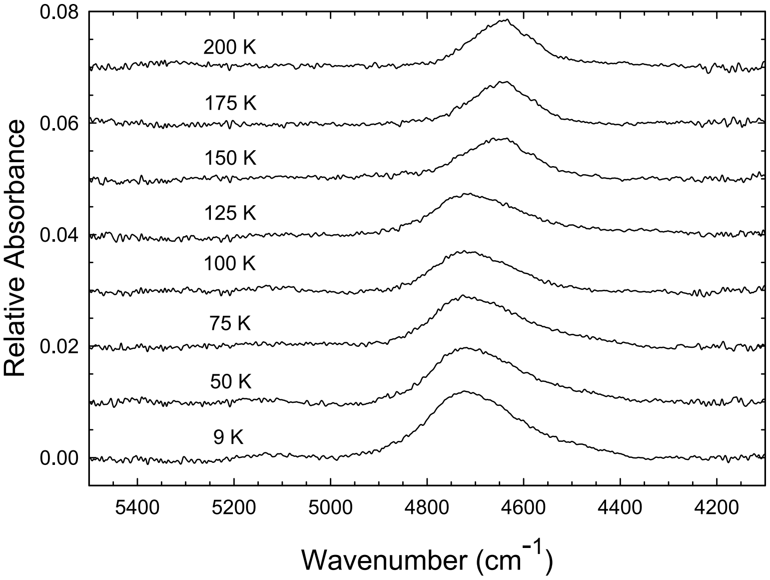

FIG. 2. Warming sequence for a pure H_2O_2 ice.

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 GiguËre, 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

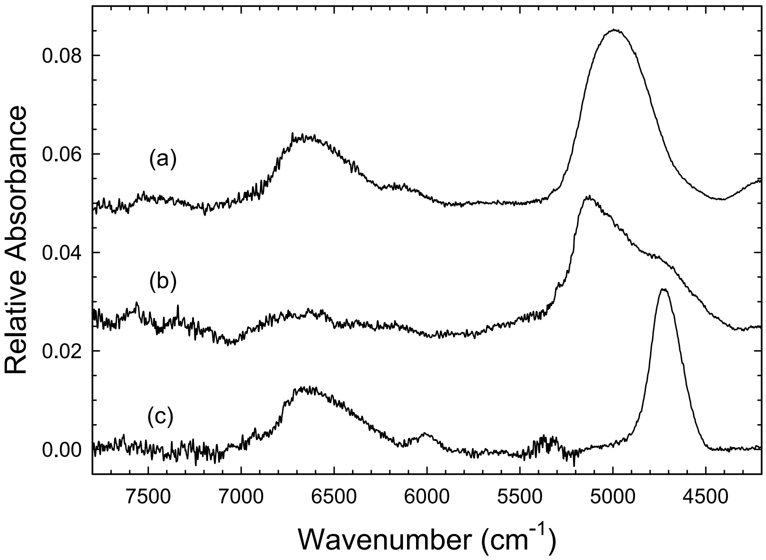

FIG. 3. Near-IR spectra at ~10 K for samples with three different H_2O_2 concentrations: (spectrum a) pure H_2O, (spectrum b) 30% H_2O_2, and (spectrum c) 100% H_2O_2.

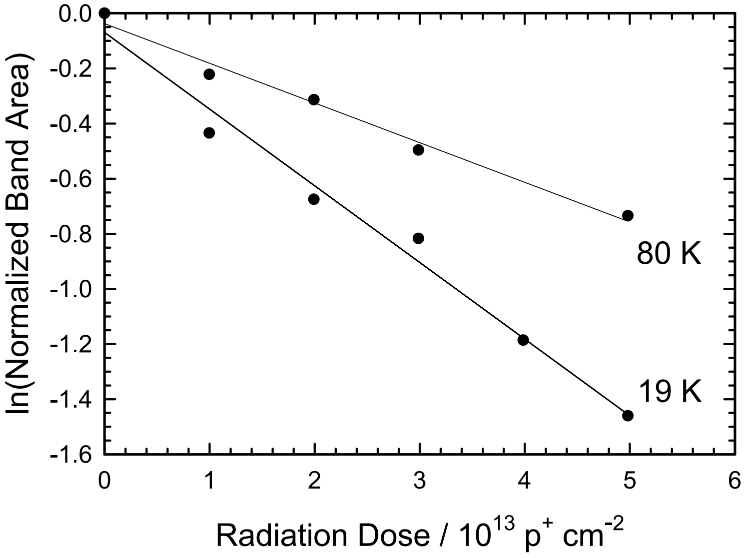

FIG. 4. Radiation-induced decay in 3% H_2O_2 by 0.8 MeV H^+.

H_2O_2. 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:

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

Here *N* is the column density of the sample, in molecules/cm^2, and $\int \tau(\tilde{v})\, d\tilde{v}$ 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 $A(H_2O_2, 2850\ cm^{-1}) = 2.7 \times 10^{-17}$ cm/molecule for a 3% $H_2O_2 + H_2O$ mixture (Moore and Hudson, 2000). Using this *A* value, we find $A(H_2O_2, 4725\ cm^{-1}) = (2.7 \times 10^{-17}\ cm/molecule)/6.5 = 4.2 \times 10^{-18}$ 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 H_2O and H_2O_2 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 $A(H_2O_2, 10\ K, 4725\ cm^{-1}) = 4.2 \times 10^{-18}$ cm/molecule is the same order of magnitude as $A(H_2O, 10\ K, 5040\ cm^{-1}) = 1.2 \times 10^{-18}$ cm/molecule recently reported for an overtone of H_2O (Gerakines *et al.*, 2005).

Recently, Loeffler *et al.* (2006) measured $A(H_2O_2, 80\ K, \sim 2850\ cm^{-1}) = 5.2 \times 10^{-17}$ 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 $A(H_2O_2, 4725\ cm^{-1}) = (5.2 \times 10^{-17}\ cm/molecule)/6.5 = 8.0 \times 10^{-18}$ cm/molecule. The techniques and H_2O_2 sources for obtaining these two $A(H_2O_2)$ 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 H_2O_2 and H_2O. The strongest bands for pure H_2O_2 (4725 cm^{-1}) and pure H_2O (5000 cm^{-1}) in Fig. 3 do show differences in position and profile. For the small H_2O_2 abundances that occur on Europa and are expected to occur on Mars and in the outer Solar System, however, the near-IR spectra of $H_2O_2 + H_2O$ mixtures are essentially the same as those of pure H_2O. We conclude that the prospects for using the 7500–4500 cm^{-1} region to study H_2O_2 in H_2O-dominated ices are poor.

In our earlier work we showed that the equilibrium H_2O_2 abundance reached in 800 keV H^+-irradiated H_2O-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^+ (Loefler *et al.*, 2006) and 200 keV H^+ (Gomis *et al.*, 2004b). However, it has also been found (Gomis *et al.*, 2004a) that the H_2O_2 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 H_2O_2 IN H_2O-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: 1.0×10^{13} p^+ cm^2 incident = 43.9 Mrad = 0.439 MGy = 0.082 eV/H_2O molecule = 0.092 eV/16-amu molecule.

[a] A 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 H_2O_2 paper (Moore and Hudson, 2000), we also described how scavenging experiments suggest that electron-transfer reactions are important for explaining the differences in H_2O_2 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 H_2O_2 abundance at the higher temperature might be due to a greater rate of H_2O_2 destruction. In the present work, H_2O_2 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% H_2O_2 ice to be the same as in pure H_2O-ice. This value was calculated to be 274 MeV cm^2/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: 1.0×10^{13} p^+ cm^2 incident = 43.9 Mrad = 0.439 MGy = 0.082 eV/H_2O 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 $t_{1/2} = -\ln(2)/\text{slope}$ 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 H_2O_2 destruction might be faster at the higher temperature is contradicted by the data summarized in Fig. 4 and Table 1. The smaller H_2O_2 abundance seen (Moore and Hudson, 2000) after H_2O-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 H_2O_2 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 H_2O_2 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.*, H_2O). Since H_2O_2 *is observed* at the present, then its efficient formation is suggested, aided either by the presence of other molecules within the irradiated H_2O-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 H_2O_2 + H_2O-ices show-

TABLE 2. HALF-LIVES FOR H_2O_2 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

[a] Assumes an ice density of 1 g/cm^3.

[b] From Fig. 16 of Cooper *et al.* (2001).

[c] Using $t_{1/2}$ (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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