

Laboratory Reflection Spectra for the Determination of Chemical Composition of Icy Bodies

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The visible (0.3 to 1.03 μm) spectral reflectances of H_2O , NH_3 , H_2S , and NH_4HS frosts and mixtures of these frosts and their uv irradiated products have been measured. These spectra are compared with the spectra of the Galilean satellites of Jupiter, Saturn's rings and Saturn's satellites to place limits on their surface composition.

INTRODUCTION

Investigations of the ir reflectance spectra (1.25–2.5 μm) of the Galilean satellites and the rings of Saturn (Filcher *et al.*, 1972; Fink *et al.*, 1973; Kieffer and Smythe, 1974; Pilcher *et al.*, 1970; Kuiper *et al.*, 1970) have shown that Saturn's rings and the surfaces of J2 and J3 and possibly J4 are composed mainly of water frost. But, the visible (0.3–1.1 μm) spectral reflectance of these bodies decreases toward the blue and uv (Lebofsky *et al.*, 1970; Johnson and McCord, 1970) which is inconsistent with the "white" spectral reflectance of pure water frost. On the surfaces of J2 and J3, water frost and ammonium hydrosulfide (NH_4HS) frost and the uv irradiation products of H_2S and NH_4HS [H_2S_x and $(\text{NH}_4)_2\text{S}_x$] are probably stable (against evaporation) over the age of the solar system, while at the distance of Saturn, NH_3 frost also becomes stable (Lebofsky, 1975). As a result of consideration of the formation conditions of low temperature condensates (Lewis, 1971, 1972), these

frosts have been considered likely candidates to be found in the outer solar system, and hence have been chosen for laboratory study.

METHOD

The apparatus used in these experiments has previously been described in detail by one of the authors (Lebofsky, 1973). Pure gases or mixtures of gases are introduced into a vacuum chamber and deposited on a 1½ inch diameter stainless steel "cold finger", held at a temperature of 77°K. A grating monochromator (350mm focal length, 10 to 20 Å resolution) is used to scan the spectral region 0.3 to 1.03 μm . A smoked MgO surface (freshly deposited before each run) is used as a reflectance standard, and every spectrum presented is the ratio of the frost spectrum to that of the MgO standard, normalized to a maximum of 1.0. All experiments are done with a 45° incident light angle and at 90° phase angle.

For studies of uv irradiated samples, a xenon arc lamp (Christie Electric Corp CXL-300 HL) is used to irradiate the frost sample surface before being scanned with the monochromator. The 300-watt xenon arc lamp has a spectral energy distribution similar to that of the Sun, but with a cutoff

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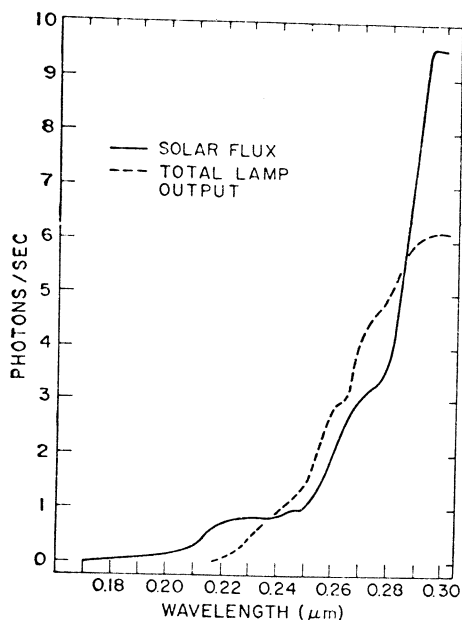


FIG. 1. A comparison of solar flux and lamp output. The solar flux is in 10^{12} photons cm^{-2} $\text{\AA}^{-1}\text{sec}^{-1}$ at a distance of 1 AU from the Sun. The lamp flux is the total output in 10^{15} photons $\text{\AA}^{-1}\text{sec}^{-1}$, of the lamp. (Determined from the lamp output figures supplied by the Christie Electric Corp. and the transmission function of the optical system).

at about $0.22\mu\text{m}$ (Fig. 1). A quartz lens is used to focus the lamp on a circular area on the "cold finger": about $1\frac{1}{2}$ inch in diameter. One hour of irradiation approximately equals 15 and 58 days of solar irradiation at the distances of Jupiter and Saturn, respectively.

The gas is deposited as a frost at a very slow rate (about 1 hour) to a thickness of about 2–5 mm with an average particle size of about $10\text{--}100\mu\text{m}$. Matheson C. P. Grade H_2S and Matheson Anhydrous NH_3 are used in all experiments. In experiments using water frost, distilled water is boiled under vacuum and the vapor allowed to condense on the surface of the "cold finger".

RESULTS²

H_2O . Figure 2 shows the reflection spectrum of H_2O frost, unirradiated. The

² All figures are hand-smooth curves smoothed to eliminate scatter in the data (less than $\pm 2\%$).

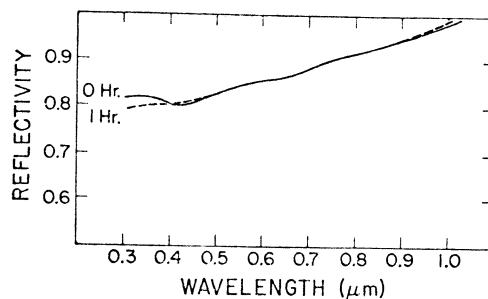


FIG. 2. H_2O frost reflection spectra.

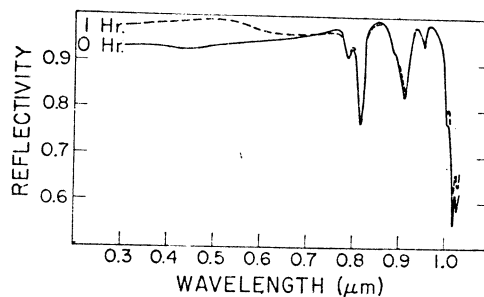


FIG. 3. NH_3 frost reflection spectra.

spectrum is featureless except for a slight increase in reflectivity toward the ir. One hour of irradiation does not produce any significant change in the spectral reflectivity of the water frost.

NH_3 . The reflection spectrum of unirradiated NH_3 frost (Fig. 3) does show some spectral features in the near ir, probably due to solid phase vibrational absorptions. The rest of the reflection spectrum is relatively featureless. Irradiation of the frost does not significantly change the spectral reflectance.

$\text{NH}_3 + 10\text{H}_2\text{O}$. The spectral reflectance of a mixture of ammonia and water frost (presumably $\text{NH}_3 \cdot \text{H}_2\text{O} + 9\text{H}_2\text{O}$) is featureless and unaffected by irradiation (Fig. 4).

H_2S . The reflection spectrum of the unirradiated H_2S frost is, like the previous frost spectra, flat and featureless (Fig. 5). But, upon irradiation there is a significant change in the reflection spectrum of the

Original data have a resolution of $10\text{--}20\text{\AA}$. Each reflection curve represents one run (several averaged scans on a frost sample) which is representative of at least two or three separate experiments under similar conditions. Variations from experiment to experiment were less than $\pm 5\%$.

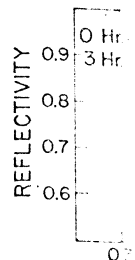
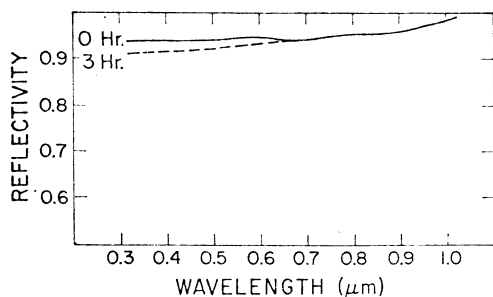


FIG. 4. $\text{NH}_3 + 10\text{H}_2\text{O}$

frost. The reflectivity is below 0.5, and increases with increasing irradiation, shifting toward longer wavelengths. $\text{H}_2\text{S} + 10\text{H}_2\text{O}$ unirradiated. $0.85\mu\text{m}$ and $0.7\mu\text{m}$, do not shift toward $0.35\mu\text{m}$.

FIG. 4. $\text{NH}_3 + 10\text{H}_2\text{O}$ frost reflection spectra.

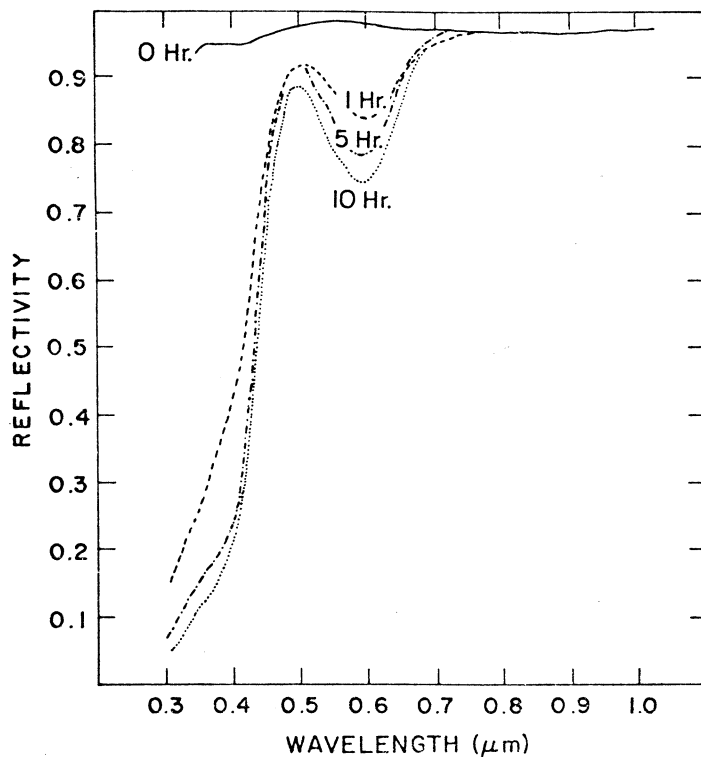
frost. There is a decrease in reflectivity below $0.5\mu\text{m}$ and there is also a broad absorption feature centered at $0.6\mu\text{m}$ which increases in strength with increasing irradiation time. The short wavelength absorption feature also appears to be increasing in strength as the band edge shifts toward longer wavelengths.

$\text{H}_2\text{S} + 10\text{H}_2\text{O}$. The reflectance of the unirradiated frost decreases longward of $0.85\mu\text{m}$ and decreases slowly shortward of $0.7\mu\text{m}$, decreasing rapidly shortward of $0.35\mu\text{m}$. Irradiation tends to weaken the

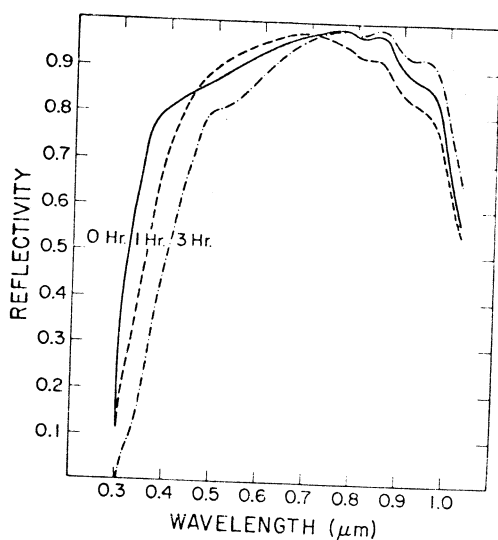
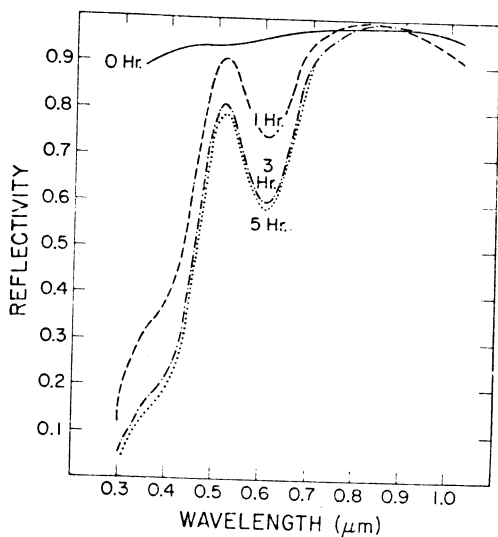
ir absorption feature but increases the strength of the short wavelength absorption feature (Fig. 6).

NH_4HS . The reflection spectrum of the unirradiated NH_4HS frost is flat and featureless (Fig. 7). Upon irradiation, the spectrum appears to be similar to that obtained by the irradiation of pure H_2S frost. Again, with increasing irradiation time the $0.6\mu\text{m}$ absorption feature increases in depth and the short wavelength absorption feature moves toward longer wavelengths. In several experiments uv cutoff filters were used to remove radiation shortward of about $0.3\mu\text{m}$ from the irradiating light. It was found that removal of the short wavelength radiation significantly reduced the irradiation effects implying that the changes produced in the frosts are due to radiation shortward of $0.3\mu\text{m}$.

$\text{NH}_4\text{HS} + \text{H}_2\text{O}$. The irradiated frost spectrum is similar to that of $\text{H}_2\text{S} + 10\text{H}_2\text{O}$ (Fig. 8). Irradiation tends to strengthen the uv absorption feature, but the ir absorption feature remains about the same. Also,

FIG. 5. H_2S frost reflection spectra.

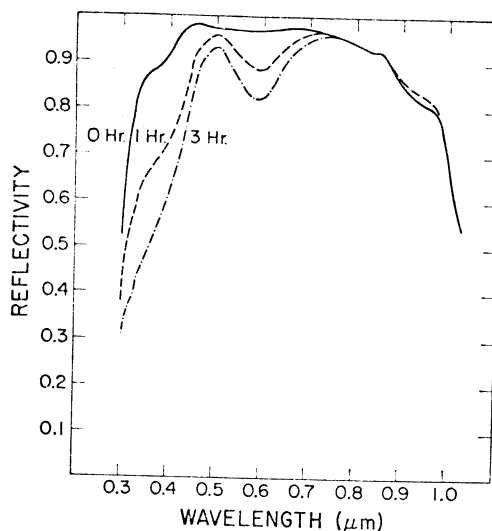
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FIG. 6. $\text{H}_2\text{S} + 10\text{H}_2\text{O}$ frost reflection spectra.FIG. 7. NH_4HS frost reflection spectra.

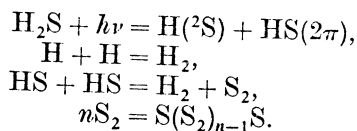
a $0.6\mu\text{m}$ absorption band appears which increases in strength with increasing irradiation.

DISCUSSION

Experimental studies on the photolysis of solid H_2S , D_2S , and H_2S_2 have been conducted by several investigators. Their results are applicable to the present work and will be used to help interpret the data obtained. Stiles *et al.* (1966) photolyzed solid H_2S , D_2S , and H_2S_2 at 77°K using light with wavelengths greater than

FIG. 8. $\text{NH}_4\text{HS} + \text{H}_2\text{O}$ frost reflection spectra.

$0.22\mu\text{m}$. Based on the electron spin resonance spectra of the radicals produced, Stiles and his co-workers proposed a mechanism similar to the gas phase mechanism in which the primary photolytic process is dissociation of H_2S to a hydrogen atom and a mercapto radical. They proposed the mechanism



This mechanism agrees with one postulated by Goldberg (1964) who photolyzed H_2S clathrate hydrate at 193°K , using the $0.2537\mu\text{m}$ Hg line and the $0.2288\mu\text{m}$ Cd line. Further reaction of the S_{2n} diradical with hydrogen atoms and mercapto radicals can give hydrogen polysulfides.

Work by Feher and Munzer (1963), Meyer *et al.* (1972a,b), Lebofsky (1973), and Fegley (1974) indicates that two different sulfur species are responsible for the absorption bands observed in irradiated H_2S and NH_4HS frosts. Feher and Munzer (1963) have taken the uv absorption spectra of hydrogen polysulfides H_2S_x (where $x = 1-8$) from 0.20 to $0.45\mu\text{m}$. Polysulfides with $x = 5-8$ all exhibit absorption shortward of $0.45\mu\text{m}$. The shorter chain polysulfides do not begin absorbing until farther into the uv region.

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The work by Meyer *et al.* (1972b) and Meyer and Spitzer (1972) suggests that sulfur radicals may be responsible for the $0.6\mu\text{m}$ visible absorption band observed by Lebofsky. Their experiments and extended Hückel calculations show that the absorption of a diradical sulfur chain moves to longer wavelengths as the chain length increases. S_2 trapped in different matrices at 4°K and 20°K and in an isopentane-methylcyclohexane (IPC) glass at 77°K absorbs around $0.40\mu\text{m}$, and S_4 in an IPC glass absorbs at $0.53\mu\text{m}$. According to the Hückel calculations, absorption around $0.6\mu\text{m}$ is due to S_5 - S_7 sulfur diradicals.

From these results it would appear that the uv dropoff observed in the spectra of uv irradiated H_2S frost and clathrate hydrate is probably due to electronic transitions in H_2S_5 - H_2S_8 polysulfides while the absorption band at $0.6\mu\text{m}$ is probably due to transitions in sulfur diradicals, S_5 - S_7 . For the irradiated NH_4HS frost, the short wavelength absorption is probably due to ammonium polysulfides $(\text{NH}_4)_2\text{S}_x$ (where $x = 5-7$).

The decrease in reflectivity in the near ir for $\text{H}_2\text{S} + 10\text{H}_2\text{O}$ and $\text{NH}_4\text{HS} + \text{H}_2\text{O}$ (Figs. 6 and 8) is unexplained but may be due to formation of the hydrated forms of these frosts. H_2S forms a type I clathrate hydrate with water with a maximum composition of $\text{H}_2\text{S} \cdot 5.7\text{H}_2\text{O}$, although NH_4HS does not. The unit cell of the structure I hydrate is composed of 46 water molecules enclosing 2 small cavities of 5.1Å diameter and 6 larger cavities of 5.8Å diameter. NH_4HS may form a type II clathrate hydrate with water, although this has not been reported. The structure II hydrate unit cell is characterized by 136 water molecules, 16 small cavities (approximately 5.0Å in diameter) and 8 larger cavities of 6.7Å diameter (Mandelcorn, 1959; van der Waals and Platteeuw, 1959). The local symmetry fields that the H_2S and NH_4HS molecules experience in the clathrate cavities may result in different symmetry transformations of their molecular orbitals and vibrational modes than in the H_2S and NH_4HS frosts. Thus, different spectra would result in the hydrates than in the frosts. Further studies of the

reflection and absorption spectra of H_2S , NH_4HS , and the corresponding hydrates in the region $0.7-1.5\mu\text{m}$ will be necessary to determine if this interpretation is correct.

CONCLUSIONS

Saturn's satellites have been observed only in the visible ($0.3-1.1\mu\text{m}$) (McCord *et al.*, 1970). These satellites have flat, featureless reflection spectra so that no positive determination of composition is possible. From the results of the frost stability calculations (Lebofsky, 1975) and the laboratory spectral data, the possible frosts present on the surfaces of the satellites are ammonia, ammonia hydrate and water frosts. The sulfur-bearing frosts have reflection spectra which are featureless before irradiation, but because there is no way to prevent the irradiation of the frosts on the surfaces of Saturn's satellites, we must base our conclusions on the properties of uv-irradiated frosts. Since the sulfur-bearing uv-irradiated frosts have

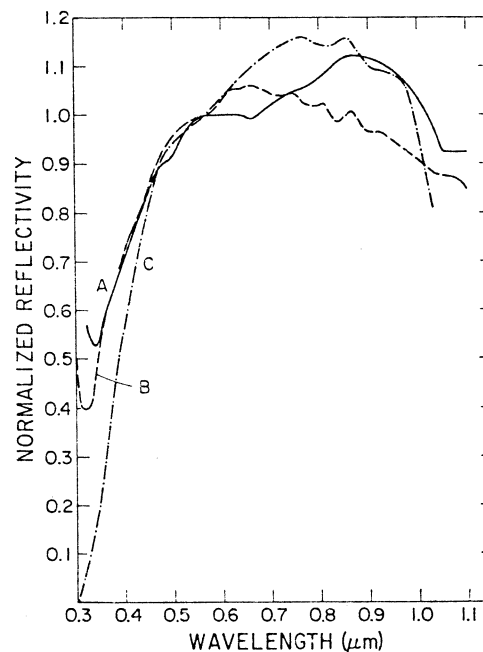


FIG. 9. Normalized reflectivity ($0.566\mu\text{m}$) of $\text{H}_2\text{S} + 10\text{H}_2\text{O}$ compared with J3. A: J3 (Johnson and McCord, 1971); B: J3 (Wamsteker, 1972, 1975); C: $\text{H}_2\text{S} + 10\text{H}_2\text{O}$, 3 hours irradiation

blue and uv decreases in their reflectivity, they can be eliminated as possible *major* surface components of Saturn's satellites. Both laboratory ir (1-4 μ m) reflection spectra of uv irradiated frosts (H₂S, NH₄HS, H₂S + 10H₂O and NH₄HS + H₂O) and telescopic ir (1-4 μ m) spectra are needed to determine more accurately the composition of these satellites.

Saturn's rings and the Galilean satellites J2 and J3 have ir reflection spectra consistent with the presence of water frost (Pilcher *et al.*, 1970; Pilcher *et al.*, 1972; Fink *et al.*, 1973; Kieffer and Smythe, 1974), but their visible spectra are inconsistent with the presence of pure water frost (Johnson and McCord, 1970; Lebofsky *et al.*, 1970). Based on ir spectra, J4 may have some H₂O frost on its surface, but because of its low albedo silicates and/or carbonaceous chondritic material may be

the major component on its surface (Johnson and Pilcher, 1974). J1, although it has a high albedo, does not appear to have any surface water frost (Pilcher *et al.*, 1972; Fink *et al.*, 1973; Kieffer and Smythe, 1974). However, it may have evaporite minerals and/or sulfur on its surface (Fanale *et al.*, 1974, 1976; Wamsteker *et al.*, 1974). Lebofsky (1973) and Wamsteker (1974, 1975) independently concluded that polysulfide contamination of H₂O frost might explain the reflection spectra of J2, J3 and Saturn's rings.

Figure 9 compares the visible reflection spectra of (Johnson and McCord, 1971; Wamsteker, 1972, 1975) with the laboratory reflection spectrum of H₂S + 10H₂O. The curves for J3 (Johnson and McCord, 1971) and Saturn's ring B (Lebofsky *et al.*, 1970; see Fig. 10) have been modified because of improvement in filter calibra-

TABLE I

Wavelength	Corrected wavelength	Old ^a JIII	Old ^b Ring B	Correction ^c	Fig. 9 New JIII	Fig. 10 New Ring B
0.319	0.326	0.644	0.394	0.884	0.569	0.348
0.338	0.343	0.577	0.432	0.909	0.524	0.392
0.358	0.360	0.589	0.428	1.023	0.602	0.437
0.383	0.383	—	0.472	1.048	—	0.495
0.402	0.403	0.771	0.623	0.911	0.702	0.568
0.433	0.435	0.876	0.715	0.915	0.802	0.654
0.467	0.469	0.924	0.799	0.969	0.895	0.774
0.498	0.500	0.954	0.894	0.955	0.912	0.854
0.532	0.534	0.997	0.971	0.985	0.982	0.956
0.564	0.566	1.000	1.000	1.000	1.000	1.000
0.598	0.599	1.016	1.043	0.984	1.000	1.026
0.633	0.633	1.028	1.094	0.976	1.003	1.068
0.655	0.655	1.024	1.097	0.970	0.994	1.064
0.699	0.699	1.020	1.099	1.000	1.020	1.099
0.730	0.730	1.028	1.117	1.013	1.041	1.131
0.765	0.764	1.036	1.121	1.017	1.053	1.140
0.809	0.806	1.052	1.114	1.028	1.081	1.145
0.855	0.855	1.096	1.150	1.029	1.122	1.183
0.906	0.905	1.109	1.174	1.002	1.111	1.176
0.948	0.947	1.138	1.177	0.954	1.085	1.123
1.002	1.002	1.072	1.155	0.968	1.037	1.118
1.053	1.053	0.979	1.210	0.945	0.925	1.143
1.101	1.098	0.967	1.090	0.955	0.924	1.041

^a Johnson and McCord (1971).

^b Lebofsky *et al.* (1970).

^c Chapman *et al.* (1973).

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tion and standard star fluxes (Chapman *et al.*, 1973; see Table I). The $0.6\mu\text{m}$ feature appears only as a shoulder, matching very well the shoulder in the spectra of the solar system bodies. The match in the region of the uv feature and the ir feature is good considering the match of the two J3 spectra in the near ir (Johnson and McCord, 1971; Wamsteker, 1972, 1975). The exact strength of the $0.6\mu\text{m}$ feature relative to the uv feature depends greatly on the temperature of the surface. However, the amount of H_2S and NH_3 in the water frost will affect the strength of the feature. The spectrum of $\text{NH}_4\text{HS} + \text{H}_2\text{O}$ frost may also match the spectra of the satellites and rings, depending on the exact temperature and composition of the frost (Lebofsky, 1973).

It was very difficult to control the rate of deposition of gases with water vapor on the cold finger. Estimates of composition were based on the total amount of frost deposited and the amount of gas used. In general, the $\text{H}_2\text{S}/\text{H}_2\text{O}$ ratios were approximately 1:10 while the $\text{NH}_4\text{HS}/\text{H}_2\text{O}$ ratio was about 1:1. Therefore, the strength of the $0.6\mu\text{m}$ absorption feature is probably dependent on the degree of dilution with H_2O . Thus, a spectrum of irradiated $\text{NH}_4\text{HS} + 10\text{H}_2\text{O}$ would probably have a shoulder at $0.6\mu\text{m}$ and look more similar to the $\text{H}_2\text{S} + 10\text{H}_2\text{O}$, satellite, and ring spectra.

Also, preliminary results (Lebofsky, 1973) indicate that the strength of the $0.6\mu\text{m}$ absorption band is temperature dependent, becoming weaker as the temperature increases above 77°K . This effect is irreversible, and is due to faster radical diffusion and recombination in a matrix at higher temperatures. It is expected (by analogy with spectra of orthorhombic S_8) that the position of the uv dropoff in the reflection spectra will reversibly change with temperature. The shift of the uv absorption edge of orthorhombic S_8 is reversible and is caused by a change in thermal population of the vibrational levels of the electronic ground state of S_8 (Meyer *et al.*, 1972a). The uv absorption shift in S_8 has a magnitude of $2.3 \times 10^{-4} \mu\text{m}/^\circ\text{K}$ and the expected shift in

the frost reflection spectra may be of the same magnitude. Wamsteker (1974) has observed a similar effect.

In Fig. 10 we compare the $\text{H}_2\text{S} + 10\text{H}_2\text{O}$ spectra with spectra of Saturn's rings (Lebofsky *et al.*, 1970; Wamsteker, 1975; Irvine and Lane, 1973). Again, in the near ir there is disagreement among the ring spectra, but the overall trends are similar. The large variations between the ring spectra may be due to the higher resolution of Wamsteker and the method of Irvine and Lane which may not have completely removed the contribution of Saturn (Lebofsky, 1973).

NH_4HS and H_2S hydrate and their uv irradiated products are probably stable enough to exist on the surfaces of the Galilean satellites and in the rings of Saturn (Lebofsky, 1975). Based on these stability considerations and on the comparisons of visible and infrared reflection

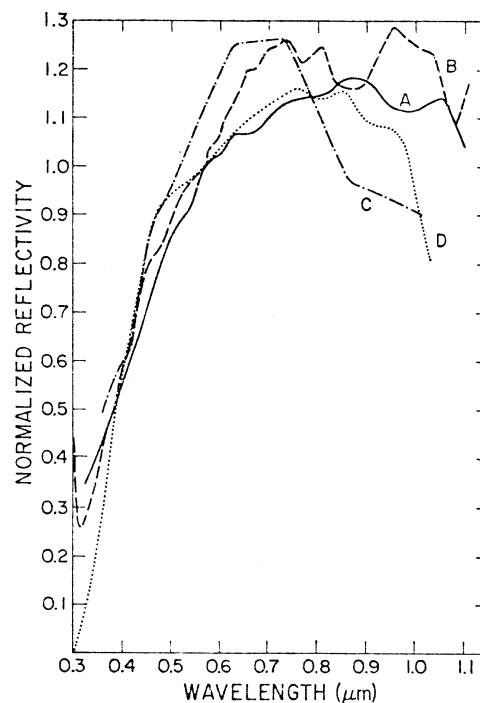


FIG. 10. Normalized reflectivity ($0.566\mu\text{m}$) of $\text{H}_2\text{S} + 10\text{H}_2\text{O}$ compared with Saturn's rings. A: Ring B (Lebofsky *et al.*, 1970); B: Ring B (Wamsteker, 1975); C: Rings (Irvine and Lane, 1973); D: $\text{H}_2\text{S} + 10\text{H}_2\text{O}$, 3 hours irradiation.

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spectra, we can conclude that the uv irradiated products of H_2S and NH_4HS are likely contaminants of the water frost present on the surfaces of the Galilean satellites and in the rings of Saturn.

The identification of these contaminants is not conclusive. No investigations have been carried out to determine the ir (1-4 μm) spectral reflectivity of irradiated contaminated water frost or of unirradiated water frost contaminated with sulfur species. Greater dilution may be possible without affecting the overall reflection spectra of the contaminated frosts. It is unknown, though, what the ir reflection spectra of these contaminated and irradiated water frosts look like or how much contaminant can be added to water frost without affecting the ir reflection spectrum of the water frost. Until ir spectra of contaminated and irradiated water frosts can be investigated and compared with the spectra of Saturn's rings and the Galilean satellites, we cannot make any firmer conclusions as to the composition of these objects.

It should be noted that we have been dealing here with pure frosts. No attempt has been made to study the effects of mixing silicates with the frosts or dusting the surfaces of the frosts with silicate materials. As has been previously stated, the reflectivities of most of the satellites and Saturn's rings are high and so the surfaces of these objects are composed predominantly of frosts. The addition of small amounts of silicate material will have little effect on the spectral reflectivities of frosts (unless the silicate forms a dust layer on the frost or is fine enough to form a "dirty" frost) and so would have little effect in our comparisons of laboratory frosts and icy surfaces.

ACKNOWLEDGMENTS

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