

that in SSBD (1), even though the hydrogen bonding is completely different—in the latter structure there are no intramolecular hydrogen bonds. The only significant difference, about 10°, is in the conformation about the C₁—O₁ bond. Conformations about the bonds C₅—C₆ and C'₅—C'₆ are also very nearly the same in sucrose and in SSBD. However, the conformation about the bond C'₂—C'₁ differs by 135° between the two structures.

There is a significant difference in the conformation of the furanose ring between sucrose and SSBD. In the SSBD crystal, atoms C'₂, C'₃, C'₅, and O'₂ are very nearly coplanar, and atom C'₄ is about 0.5 Å from their average plane. The furanose ring in the sucrose crystal cannot be described so simply. We prefer to describe it by specifying an angle of twist about each ring bond. Consider four successive atoms around the ring, as C'₅, C'₄, C'₃, C'₂. We define the conformation angle of the directed bond C'₄→C'₃ as the angle, measured counterclockwise, that the projection of bond C'₄→C'₅ makes relative to the projection of bond C'₃→C'₂ when one looks in the direction of the bond C'₄→C'₃. In the furanose ring of sucrose the conformation angles for the various bonds are: C'₄→C'₃, 34.9°; C'₅→C'₄, -27.4°; O'₂→C'₅, 8.3°; C'₂→O'₂, 14.5°; C'₃→C'₂, -31.0°. Corresponding values for SSBD are 36.5°, -34.5°, 22.9°, -1.5°, and -21.1°.

The pyranose ring, as in SSBD, is in the chair form, with —CH₂OH and —OH groups in equatorial positions and O₁ and 5 hydrogen atoms in axial positions. The six conformation angles of the ring, alternating in sign, fall within the range of magnitude 54.5° to 55.5°. The range is wider, 51.3° to 64.0°, in SSBD, probably reflecting the larger coordinate errors.

Lengths (12) of C—C bonds (Fig. 2) show remarkably small deviations from the mean of 1.52 Å, regardless of environment. The short C—C bonds, averaging 1.44 Å, reported by Beevers and Cochran, in the furanose ring in SSBD can hardly be correct. Variations of length among the C—O bonds are slightly greater. In each ring, the C—O bond adjacent to the glycosidic linkage appears significantly shorter than the other C—O bond of the ring. The C—H bond lengths range from 1.08 to 1.11 Å; the average, 1.10 Å, is the normal value expected for saturated molecules.

The O—H bond lengths show the largest range of values, probably because of perturbing effects of hydrogen bonding and because of widely varying effects of thermal motion on apparent bond lengths. For the O₄ hydroxyl group, thermal motion has an especially large effect, since this group is not clamped in orientation by hydrogen bonding, and torsional motion about the C₄—O₄ bond is relatively free.

The apparent bond length of 0.89 Å was approximately corrected (13) for the thermal motion to 0.94 Å, on the assumption that the H atom “rides” the oxygen atom. We have not yet analyzed the thermal motion in enough detail to make corrections for other bonds.

The oxygen valence angles in the ether linkages are: C₁—O₁—C'₂ (glycosidic linkage), 114.6°; C₁—O₅—C₅, 116.0°; C'₂—O'₂—C'₅, 111.5°. Carbon valence angles inside the pyranose ring range from 108.2° to 111.2°; inside the furanose ring, from 102.2° to 105.8°. Other carbon valence angles range from 105.0° to 116.0°. Oxygen valence angles in the hydroxyl groups range from 104.6° to 111.6°.

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 10. A table of coordinates will appear in the complete account of this work to be published later. It will be furnished earlier to anyone interested.
 11. In least-squares refinement for space group $P2_1$, the y coordinate of one atom must be held constant to fix the origin of the coordinate system. We have fixed the y coordinate of atom C₁ at the value reported by Beevers *et al.* (3). The coordinate shifts given must be considered with this constraint in mind.
 12. Bond lengths are reported to two decimal places only, because precise values of the unit-cell parameters are not yet available.
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- * Oak Ridge National Laboratory is operated by Union Carbide Corporation for the U.S. Atomic Energy Commission.

24 May 1963

Interpretation of the 3- to 4-Micron Infrared Spectrum of Mars

Abstract. *Infrared reflection spectra have been recorded for a large number of inorganic and organic samples, including minerals and biological specimens, for the purpose of interpreting the 3- to 4- μ spectrum of Mars. A previous suggestion that the Martian bands indicated the presence of carbohydrates is shown not to be a required conclusion. However, no satisfactory explanation is advanced and the problem remains unresolved.*

The nature of the Martian surface and the possibility of life existing there has for decades stimulated considerable speculation and research. A notable contribution to the rather meager quantitative data on the planet was made recently by Sinton (1, 2) when he observed “absorption bands” in the 3- to 4-micron radiation reflected by the dark areas which were less pronounced in the radiation from the light areas. The spectral curves he obtained with the 200-inch (508 cm) telescope at Mt.

Palomar Observatory are shown in Fig. 1. Because of the low available energy, the resolution on the disc was poor, being at best half a planetary diameter, and the accepted area for the nominal Syrtis Major spectrum, in fact, included large expanses of bright areas. The associated spectral resolution calculated from the slit width and dispersion was 90 cm⁻¹ (0.11 μ) for some of the recordings and 45 cm⁻¹ (0.056 μ) for the remainder.

The significant observation was of a

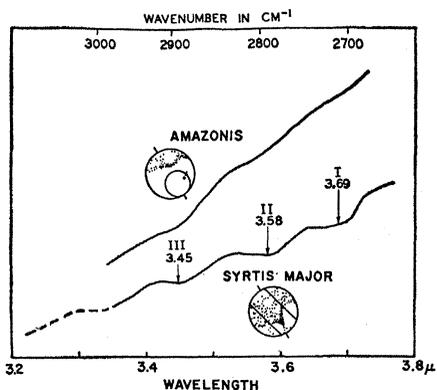


Fig. 1. The reflection spectra of Mars as obtained by W. M. Sinton. The curves shown are the result of dividing the Martian spectra by the spectrum of the sun. The inserts indicate the size and location of the aperture on the disk.

structure, more pronounced for the dark areas than for the bright ones. Sinton considered the structure to be absorption bands and to result from surface components. The bands, located at 2710 (3.69μ), 2793 (3.58μ) and 2900 cm^{-1} (3.45μ), fall in the region where organic molecules, and some inorganic compounds, absorb. Inorganic carbonates were eliminated because of a lack of spectral correspondence, but the possibility that some other inorganic compound may be responsible was not discarded. However, Sinton considered the spectra to resemble closely those of organic molecules and of plants. The

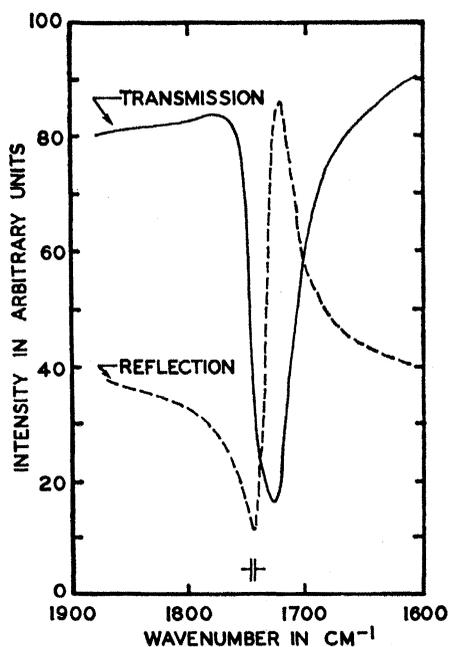


Fig. 2. The transmission and reflection spectra of the C=O band of lucite. A film 25μ thick was observed in transmission; a block 1 cm thick, in reflection.

presence of carbohydrates was inferred from the band at 2710 cm^{-1} , which he stated is present for this class of compounds and for the alga *Cladophora*. While not totally rejecting an inorganic interpretation, Sinton evidently believes this is a remote possibility and that "the presence of large organic molecules is indicated. . . ."

A variant to this interpretation has been advanced by Colthup (3) who noted that the band at 2710 cm^{-1} is unique for a C-H group in having such a low wavenumber. Not only is this band rare in plants, as Sinton commented, it is also very uncommon for organic compounds in general. The only structural component having an intense band at this wavenumber is the C-H of an aldehyde group. From the high relative intensity of this band in the Martian spectra there must be a high concentration of aldehyde C-H in relation to CH associated otherwise. The only molecule which is suitable is acetaldehyde, which also has a second band that could be contributing to the 2793 cm^{-1} absorption. The remaining 2793 cm^{-1} absorption and all of that at 2900 cm^{-1} is then due to other CH groups of the Martian organic matter. Colthup speculates that "acetaldehyde may be an end product of certain anaerobic metabolic processes." However it has been pointed out (4) that the high volatility of this chemical would ensure a high concentration in the atmosphere, so that the observed bands would have to be due to gaseous acetaldehyde. It should then be observed over the entire disk and not be restricted to certain areas, except for an unlikely situation giving rise to differences in concentration resulting from differential rates of formation and decay over the bright and dark areas.

The implications of Sinton's observations and the interpretations are clearly quite great. The potential demonstrated for infrared probing of the Martian surface is such that an infrared spectrometer has been proposed for inclusion in spacecraft experimental packages destined for Mars. We have recorded laboratory reflection spectra on a wide variety of terrestrial objects in order to facilitate interpretation of the Martian observations. We have also examined the nature and the interpretation of Sinton's observed spectra.

Without considering a comprehensive theory which would relate an observed planetary reflection spectrum to the absorption frequencies and mor-

phology of the surface material, there is one point not usually appreciated that must be stressed.

There is a fundamental difference in appearance between absorption and reflection spectra. The reflection spectrum corresponding to a simple absorption band will have a minimum on the high wavenumber side of the resonant frequency, and a maximum which lies on the low wavenumber side for weak bands but which broadens and shifts to the other side as the intensity of the associated absorption band increases. The relation between absorption and reflection for a single strong organic band is shown in Fig. 2. The transmission measurement was made on a $25\text{-}\mu$ film of lucite. A thick block of the plastic, with essentially zero transmission and a smooth reflecting surface, was used to obtain the reflection spectrum.

Now the relationship discussed is valid only for smooth surfaces and for observations at a single reflection angle on materials thick enough to absorb completely (specular reflection). As the surface is roughened some of the radiation will pass through parts of the material before being reflected. The result will be a blend of absorption and reflection features, the exact proportions being a function of the surface configuration and the dielectric properties of the solid. When the observed intensity includes reflection at a variety of angles the sharpness of the relationship between the S-type reflection curve and the absorption band is further blurred. Materials of finite optical thickness will exhibit unique effects because of reflection at the second interface. These complications render the interpretation of the reflection spectrum of an unknown body, unknown in both morphology and composition, ambiguous. And of course this situation applies to the Martian spectra.

With respect to Sinton's observations it is not possible to say whether the features are predominantly absorption or reflection, since the resolution was too low to produce definitive contours. As a result an additional uncertainty is introduced which, together with the estimated probable error of 16 cm^{-1} , leads to the following ranges for the associated vibrational wavenumbers— 2674 to 2726 , 2754 to 2809 , and 2824 to 2915 cm^{-1} . These will, throughout the remainder of this paper, be referred to as the Mars I, II, and III bands, respectively.

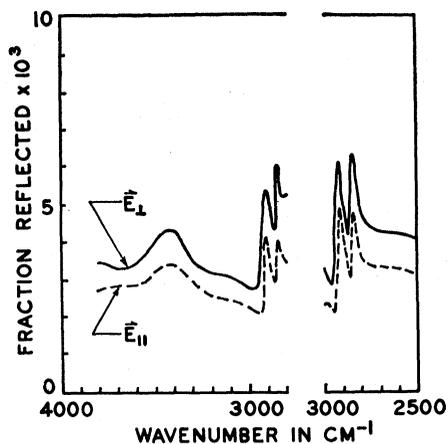


Fig. 3. The reflection spectra of an *Agapanthus* leaf for the two polarizations perpendicular and parallel to the plane of incidence. The angle of incidence was 20° . The spectra were obtained with the single beam accessory.

Because of these complexities we have not attempted to calculate these interactions from first principles but rather have determined empirically the spectral characteristics of a variety of materials under various conditions.

For the spectral observations we have used a Beckman IR-7 spectrophotometer which presents the data in the region of interest in two overlapping grating orders. In the operation of the IR-7 the slit width changes with the wavenumber in order to keep the energy incident on the detector relatively constant. Hence the spectral slit width varies with the wavenumber, for example, for the reflection spectrum of calcite in the 3rd order it increased from 23 cm^{-1} at 2500 cm^{-1} to 37 cm^{-1} at 3000 cm^{-1} , while in the 4th order it decreased from 39 cm^{-1} at 2800 cm^{-1} to a minimum of 22 cm^{-1} at 3200 cm^{-1} and then increased to 57 cm^{-1} at 4000 cm^{-1} . In the reflection spectra presented the spectral slit width is indicated only for 2900 cm^{-1} . If desired, the value for other wavenumbers can be calculated by the appropriate scaling of the calcite data.

The spectral slit widths for the transmission spectra are not shown; they were smaller than for the reflection spectra by approximately a factor of 4. Transmission measurements were made on samples prepared in a variety of ways. Powders were observed by the standard technique in which the powder is ground together with a large amount of KBr and the mixture is pressed in a die to form a translucent pellet. Algae and bacteria were deposited in thin films on a AgCl plate; lichen

such as *Evernia* were pressed into thin, rather heterogeneous, sheets, or ground with KBr and pelleted, and some samples such as the epidermis and cuticle of *Agapanthus* were examined directly. Generally no attempt was made to make the transmission measurements quantitative.

For reflection measurements we used two different experimental arrangements. In one, the standard Beckman specular reflection attachment was placed in the sample beam. All samples were examined in this manner with the angles of incidence and reflection both equal to 30° . To protect the samples from heating by the beam, and to keep unwanted radiation from striking the detector for the wide slits required, we interposed band-pass filters between the source and sample. Since the reflected energy of the sample was invariably very low the reference beam was attenuated by screens in order to obtain a convenient recorder reading. These screens were calibrated in the actual combinations used with the single-beam feature of the IR-7. Since the screens' transmissions varied with wavelength, the 100 percent lines were in effect tilted. This effect has not been factored out of the spectra presented but the appearance of the bands is still sufficiently faithful for our present purpose.

There are no quantitative reflection coefficients given since the Beckman attachment was designed to observe specular reflection only. In the case of diffuse reflection it is important to know the solid angle which is accepted by the detecting apparatus, and this is not simply calculable for this experimental arrangement. Moreover, since the slit width is not constant but varies with wavenumber, the solid angle and hence the recorded intensity is also dependent on wavenumber. The resulting spectra are hence only qualitative.

The second means of recording the reflection spectra was a single beam attachment which we constructed to enable us to observe at angles of incidence and reflection from 50° to 80° and to obtain quantitative reflection coefficients. The image of the front surface of the sample is projected on the entrance slit with $f/10$ optics so that the accepted solid angle is 7.8×10^{-3} steradians. Incorporated in the device was a film polarizer (5). Thus we were equipped to study both the intensity and polarization of the reflected radiation. Figure 3 illustrates the type of

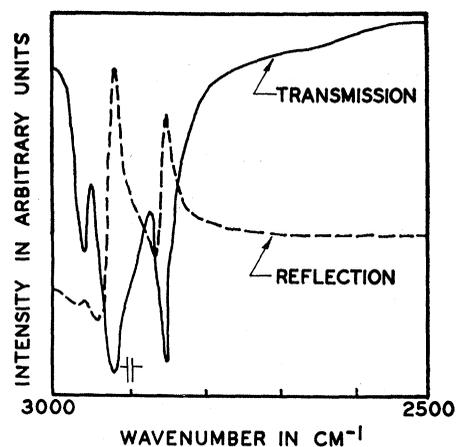


Fig. 4. The transmission and reflection spectra of paraffin wax.

quantitative reflection spectra we have obtained with this accessory at a single angle of incidence and reflection.

A large number of varying types of samples have been examined. They included pure organic chemicals, polymers, biological specimens, pure inorganic compounds, and mineralogical samples. The inorganic materials were ground to a particle size of less than 0.1 mm and then pelleted, in an attempt to produce spectra more valid for comparison with the Martian spectra than polished surfaces. No effort was made to control the particle size. The effect of this factor on the spectra is undoubtedly important and merits a careful study.

Of the many spectra recorded only a few, demonstrating specific points, will be presented here. Actually there was a considerable similarity within individual classes of compounds and the major groupings of the plants.

The reflection spectrum of paraffin

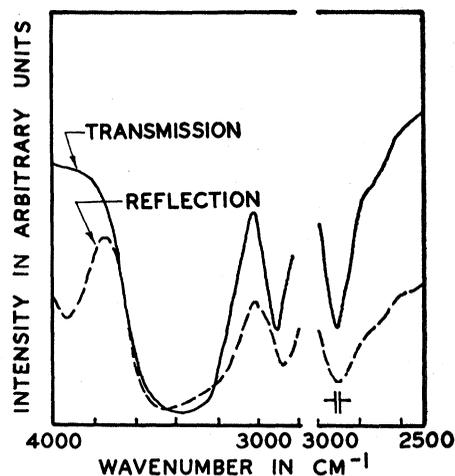


Fig. 5. The transmission and reflection spectra of cellulose.

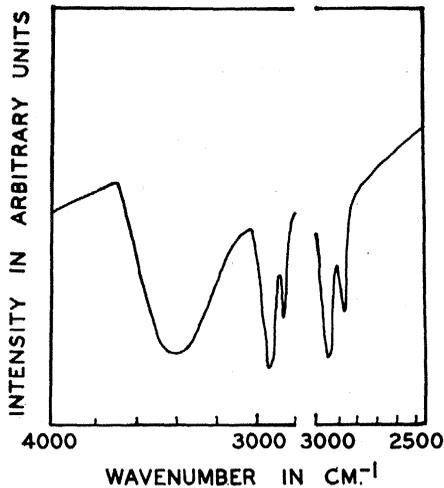


Fig. 6. The transmission spectrum of the epidermis and cuticle of an *Agapanthus* leaf.

wax, Fig. 4, is primarily a superposition of two S-type curves similar to that in Fig. 2. The energy minima of the curves are shifted about 18 cm^{-1} toward higher wavenumbers from the positions, 2850 and 2920 cm^{-1} , of the absorption bands.

Cellulose, a major constituent of plants, has a simple reflection spectrum with a strong minimum at 2900 cm^{-1} and a weak shoulder near 2750 cm^{-1} , Fig. 5. This shoulder has far too low an intensity relative to the 2900 cm^{-1} absorption to account for the Mars I or II bands since this would require a much more intense Mars III band. While the reflection from the related carbohydrates—starch and sugar, show little structure, even the absorption showed only a weak band at 2750 cm^{-1} relative to the major absorption between 2800 and 3000 cm^{-1} .

Sinton, in suggesting that the Mars

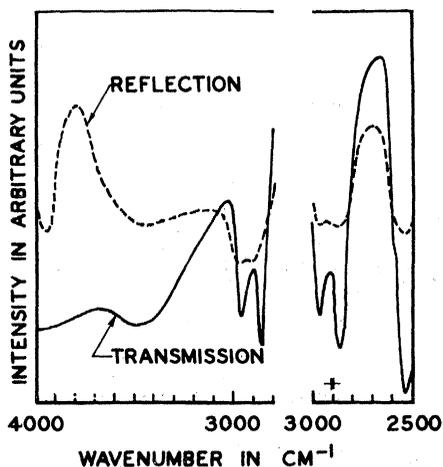


Fig. 7. The transmission and reflection spectra of calcite, CaCO_3 .

I band may be due to carbohydrates, has presented the reflection spectrum of the alga *Cladophora* which exhibits a minimum near 2750 cm^{-1} . Our spectrum of the same alga reveals only a very weak minimum at 2750 cm^{-1} with much stronger minima at 2860 and 2935 cm^{-1} . The relative intensity of the Mars I band is far too high for Sinton's interpretation and, if it is to be attributed to organic matter, it must be assigned to the C-H of free aldehyde groups. In carbohydrates such groups are present in trace amounts and would not be expected to produce intense spectral features.

Lichens have been a prime candidate for explaining some of the optical observations of Mars (6), although Salisbury (7) has recently demonstrated the weaknesses in this interpretation. Our spectra of lichen are all quite similar in having minima near 2875 and 2935 cm^{-1} , with perhaps a very faint one near 2715 cm^{-1} . The intensities are such that they could account for only the Mars III band, leaving the other two to be assigned to other surface components.

One of the most interesting biological specimens is the flat smooth leaf of the lily *Agapanthus*. The reflection spectra of both this leaf and that of the prickly-pear cactus show a marked similarity to that of paraffin wax. The reflection spectrum, Fig. 3, is characterized by two S-type curves, which correspond to the two absorption bands observed for the epidermis and cuticle in transmission, Fig. 6. For these samples the surface waxy layer is smooth enough to give nearly specular reflection, together with true reflection features.

The interest of this observation is twofold. First it demonstrates that biological objects can have reflection features of the pure reflection type as well as of the absorption type. Second, *Agapanthus* and the cactus are excellent examples of species which have a protective casing which can keep a specific component, in these instances water, from passing directly into or out of the leaf. Such membranes could be an important feature of a Martian biology.

Of the inorganic samples studied only the carbonates show much promise. Generally they have strong bands in the region 2800 to 3000 cm^{-1} , for example CaCO_3 , Fig. 7. It is relatively easy to assign to the Mars III band, since the compounds MgCO_3 , FeCO_3 ,

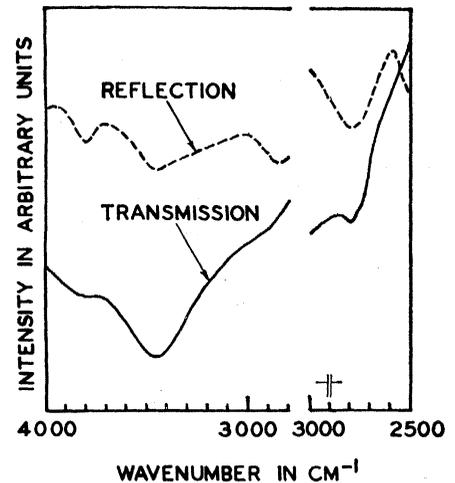


Fig. 8. The transmission and reflection spectra of PbCO_3 .

CaCO_3 , $\text{CaMg}(\text{CO}_3)_2$, ZnCO_3 , BaCO_3 , and CdCO_3 all have bands within the estimated range. The Mars II band is more difficult to interpret with only BaCO_3 , PbCO_3 , and CdCO_3 as possibilities. Now, however, we encounter problems of cosmic abundance (8). For the Mars III band only PbCO_3 , Fig. 8, comes close, with a band at 2749 cm^{-1} , but one would hardly expect a lead salt as the dominant cover over extensive areas of a planet.

The situation is then quite unsatisfactory. Assigning the Mars III band to either inorganic carbonates or organic compounds is straightforward. The other two bands remain without a satisfactory interpretation. If they are due to organic matter both must be assigned to aldehyde C-H groups, and the relative concentration must be very high. No assignment based on observed spectra has been proposed that appears

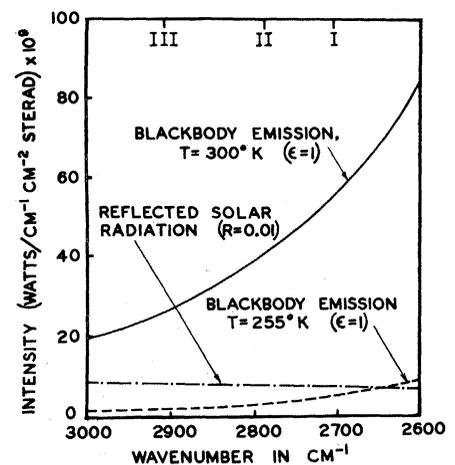


Fig. 9. Calculated intensities of the reflected and emitted radiation for a Martian surface element perpendicular to the sun.

satisfactory. On the other hand, inorganic carbonates are improbable, primarily because of the low cosmic abundance of certain necessary elements such as lead.

In his first paper Sinton (1) discussed in considerable detail the possible contributions of Martian thermal emission to the observed intensities. For the 1956 observations, for which there was no spatial resolution, he estimated an effective temperature of 279°K. After matching the experimental data with an appropriately weighted combination of a curve of a blackbody at 279°K and a laboratory reflection spectrum of lichen, he deduced that the thermal emission was a minor contribution, rising to perhaps 33 percent of the total intensity at 3.8 μ . This result was adopted in the interpretation of his 1958 observations.

To obtain a feeling for the intensities we have plotted three curves in Fig. 9—the blackbody curves for 255° and 300°K and the reflected intensity for a Martian surface element perpendicular to the sun, assuming a sun temperature of 5500°K. To calculate the latter we assumed an arbitrary reflectivity of 0.01, a value which may be somewhat low. For the emission curves an emissivity of 1 was adopted so that the values are probably a few percent high for the Martian surface. The spectra presented by Sinton (Fig. 1) are essentially effective reflectivities. However since the solar intensity varies only slightly over the wave-number range covered, they also are good representations of the energy leaving the surface.

Several points of interest now arise. It is seen that, even for a temperature of 255°K, the thermal emission can make a very significant contribution. We have not attempted to calculate effective temperatures for Sinton's two Martian spectra (Fig. 1) but we would estimate from Gifford's isotherms (9) and Sinton's 1956 calculated effective temperature of 279°K that 255°K is not excessively high. It appears then that the contribution of the thermal emission, at least near the Mars I band, is comparable to the reflected radiation.

This raises the question of the band intensities. It is expected that the emission spectrum will be relatively featureless. Whatever features are present will be opposite to the reflection features and will tend to smooth the Martian curve. It follows that the pure reflection spectrum would have spectral features

more pronounced than any observed spectrum which is the resultant of both reflection and emission. In particular, the relative intensity of the Mars I band in a pure reflection spectrum would be greater. When, in addition, one considers the large bright areas included in the nominal Syrtis Major spectrum, it is clear that the spectral features would be more intense if only the dark area were observed. At least this is true if the features are attributed to effects solely of the dark areas as Sinton's measurements indicate. This makes the assignment of the Mars I band, the one most affected by these considerations, still more difficult since we have observed in the laboratory no intense features in this region. It also stipulates that if organic matter is responsible for the other two, the material must have a very high surface concentration.

There is an alternative to the contribution of emission as an explanation for the increase of intensity in the Martian reflection in spectrum at lower wavenumbers. All of the samples examined, which contained water, show such an increase, attributable to the wing of the very intense water band at 3400 cm^{-1} . Samples containing no water, such as paraffin wax, do not exhibit this effect. If the increase in the Mars spectra is not due to the

characteristics of the emitted radiation it may arise from water in the surface material. The water could be in the liquid state in plants, in minerals as water of crystallization, or adsorbed on the surface.

We unfortunately have no answers for the major questions raised here. At present we know of no satisfactory explanation of the Martian bands. Observations of the planet with improved spectral and spatial resolution, in conjunction with radiometric temperature measurements, could possibly define the problem sufficiently to enable a solution to be found (10).

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5 June 1963

Skin Impedance and Phase Angle as a Function of Frequency and Current

Abstract. *The impedance of the human skin decreased from approximately 130 to 30 Kohm as the frequency of a-c input increased from 1 to 1000 cycles per second. Over this same range of frequencies, the phase angles changed from -2 to -58 degrees. Changing the peak-to-peak current through the subject from 14 to 62 microamperes had no effect on either the impedance of the skin or the phase angles.*

An adequate system for measuring the electrical properties of the skin, such as that required in research on galvanic skin response, should have three minimum requirements. It should (i) be relatively free from artifacts due to the apparatus, such as electrode polarization and uncontrolled variations in current; (ii) use parameters whose effects are understood; and (iii) give results which are unambiguous and meaningful within the standard body of electrical theory. The d-c systems which are commonly used for studying galvanic skin responses do not generally meet

these requirements, in that polarization effects are difficult to control, the levels of current applied are often unspecified or subject to uncontrolled variation, and the assumption is sometimes made implicitly that the body acts as a simple linear resistor.

A number of studies, such as those of Burns (1) and Grings (2), have shown that the human integument has capacitative characteristics which vary as a function of the frequency of the a-c input. However, these researchers have not measured phase angles and impedance at very low frequencies. The