

## Optical Properties of Solid and Liquid Sulfur at Visible and Infrared Wavelengths

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Knowledge of the optical constants of elemental sulfur has potential applications to Venus, Jupiter, Io, Amalthea, and the Earth. The real part,  $n$ , of the index of refraction of liquid sulfur (at 133°C) and of solid orthorhombic sulfur (at 25°C) for the wavelength range 0.4–2.0  $\mu\text{m}$  were measured ellipsometrically. The imaginary part,  $k$ , of the refractive index of liquid sulfur was obtained by transmittance measurements at the same temperature and wavelength range. The reflectance of semi-infinite slabs of solid and liquid sulfur is calculated using the measured  $n$  and  $k$  values. We confirm that sulfur melts on Io would be classified as "black" by the Voyager imaging system. © 1985 Academic Press, Inc.

In tables of the cosmic abundance of the elements, sulfur is usually listed around 10th place. Within the Solar System, allotropes of elemental sulfur and long chain sulfur compounds have been proposed for a variety of locales. On Venus, with  $\text{H}_2\text{SO}_4$  droplets and gas-phase  $\text{SO}_2$ ,  $\text{H}_2\text{S}$ , and OCS well-established, elemental sulfur has been proposed in the upper clouds (Toon *et al.*, 1982, 1984), perhaps present as a coating on sulfuric acid droplets (Young, 1983), and may be an important constituent in the lower atmosphere as well (Prinn, 1979; San'ko, 1980; von Zahn *et al.*, 1983). Indeed, the yellow coloration of Venus—apparent to the naked eye when the planet is near a cloud at sunrise or sunset—may be due to elemental sulfur and its compounds. As one of several competing models for the optical frequency chromophores in the

clouds of Jupiter, Lewis and Prinn (1970) proposed ammonium polysulfide,  $(\text{NH}_4)_2\text{S}_x$ , whose synthesis under Jovian conditions apparently requires (Sill, 1976) the aqueous ammonia clouds deduced on other grounds. Near-ultraviolet irradiation of  $\text{CH}_4/\text{NH}_3/\text{H}_2\text{O}/\text{H}_2\text{S}$  gas mixtures—possibly relevant to Jovian conditions—produces polymeric sulfur at high efficiency (Khare and Sagan, 1975).

There is little doubt that sulfur plays some role on the surface of Io because of its presence in the Io-related circumjovian toroid, and because of the identification of  $\text{SO}_2$  in the gas phase in the Ionian atmosphere. Ground-based integrated-disc spectrophotometry of Io is matched fairly well by allotropes of sulfur produced by melting and rapid quenching (Nelson and Hapke, 1978). Voyager observations of multicolored flow features on Io are suggestive of quenched sulfur melts (Sagan, 1979), especially because of the anomalous temperature/viscosity behavior of liquid sulfur. However, the issue is far from settled (Fink *et al.*, 1983; Nelson *et al.*, 1983; Pieri *et al.*, 1984; Young, 1984; Gradie and Veverka,

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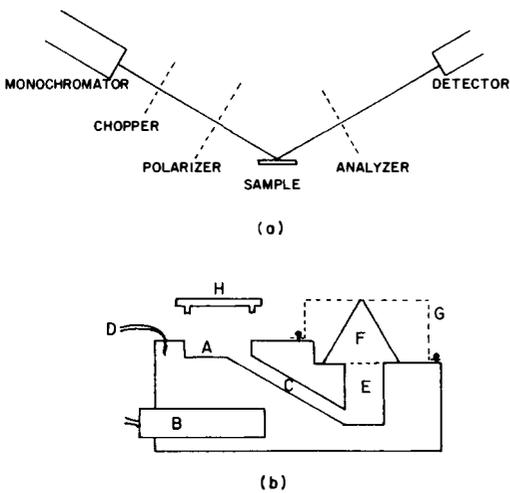


FIG. 1. (a) A sketch of the ellipsometer apparatus used for measuring the index of refraction of sulfur. (b) Aluminum cell for liquid sulfur measurements. (See text for details.)

1984; Gradie *et al.*, 1984). Liquid melts of "black" sulfur may be present in the summit calderas of some Ionian volcanoes (Sagan, 1979; Nelson *et al.*, 1983). Proton irradiation of  $\text{SO}_2$  also produces  $\text{S}_8$  (Moore, 1984). In addition, the albedo and color of Amalthea, the small Jovian satellite interior to Io, may also be due to a sulfur coating derived ultimately from Io (Gradie *et al.*, 1980, 1984).

As spacecraft exploration of the Solar System continues, there are likely to be other locales in which sulfur chemistry plays a role. In addition, of course, there are a range of terrestrial applications, including sulfur vulcanism [e.g., Watanabe (1939)], as well as biological and technological applications.

The present paper is a first-order attempt at measuring, through the visible and near-infrared spectrum, the complex refractive index of solid as well as of liquid sulfur so that radiative transfer techniques can be employed to calculate the reflectance spectrum for specified particle size distribution functions, optical depth, temperature, and other parameters. Unless laboratory conditions are identical to those in a given plane-

tary environment, simple comparison of astronomical or spacecraft observations with laboratory data can be misleading (see, e.g., Gradie and Veverka, 1984). Existing laboratory data in any way useful for such purposes are still sparse (Mondain-Monval and Schneider, 1928).

#### REFLECTION MEASUREMENTS OF LIQUID SULFUR

The ellipsometry technique is a well-known, accurate, and sensitive method for measuring the optical constants of a reflecting surface. A schematic of the apparatus used is shown in Fig. 1(a). Figure 1(b) is a sketch of the aluminum cell which we constructed for liquid sulfur measurements. The sulfur (2- to 3-mm pieces, purity 99.9995%, product of AEsar, Johnson Matthey, Inc.) was introduced into the compartment, A [Fig. 1(b)], and heated by a heating element, B, fitted into the aluminum block. The temperature was measured by an iron-constantan thermocouple, D, which was thermally fastened to the cell surface. As the sulfur melts, it flows through the tunnel, C, and gradually fills the compartment, E, while sulfur is continuously introduced into A. When the liquid begins to overflow from E, the sulfur supply is interrupted, and a quartz prism, F, is placed on the top surface of E.

An essential requirement of the measurement is a clean liquid surface kept in intimate contact with the prism (i.e., no gas bubbles at the liquid-prism interface). These conditions are accounted for as follows: (a) With the high-purity sulfur used, the liquid was free of greyish-black deposits that usually accompany molten sulfur when it is insufficiently pure. (b) Before the final fastening of the prism to the cell, it is glided upon the cell surface, back and forth, to remove gas bubbles at the interface, thus ensuring a clear liquid surface to start with. (c) To keep this interface free of gas bubbles as the measurement proceeds, the evaporation rate of the liquid is minimized by pressing the prism against the opening of E with

the aid of a clamp, G. Also, an additional amount of sulfur is introduced afterwards into A to make the liquid level in A higher, by about 2 mm, than in E. Finally, a well-fitting cover, H, closes the opening of A. With this cell, liquid sulfur at 133°C can be kept free of gas bubbles for days. Nevertheless, initial exposure to air must lead to some contamination of the liquid by SO<sub>2</sub> and H<sub>2</sub>S (Bartlett *et al.*, 1958, 1961). The liquid sulfur measured in our experiments probably contains several percent S<sub>6</sub> and S<sub>7</sub>, while the remainder is S<sub>8</sub> (Bartlett *et al.*, 1958, 1961; Steudel *et al.*, 1985).

As liquid sulfur is a good thermal insulator, the readings of the thermocouple, D, were corrected by comparing with another thermocouple whose junction point was dipped into the center of E just below the liquid level while otherwise equivalent insulating surroundings were maintained. The accuracy in the absolute values of the liquid temperature cited here is  $\pm 1.5^\circ\text{C}$ . While heating continued during the filling process, care was taken not to exceed 150°C beyond which an irreversible change in sulfur properties is noted (Nelson *et al.*, 1983), although not with very pure samples and over sufficiently long periods of time (Steudel, 1982). Before taking measurements, the temperature was kept constant for about 3 hr to allow the allotropic species to reach an equilibrium; remeasuring the sample after an overnight stay at constant temperature gave identical results.

#### REFLECTION MEASUREMENTS OF SOLID SULFUR

For solid sulfur, reflection from the free surface was measured. We used the following procedure to prepare solid sulfur: Sulfur fragments of the specified purity were placed in a beaker and heated in an oven. The molten sulfur was poured on the middle part of a preheated glass microscope slide. Two small glass spacers were placed on either edge of the glass plate. Another glass plate, either preheated or kept at

room temperature, was placed on the sulfur, thus squeezing the liquid to a thickness defined by the spacers. This sandwich was allowed to cool down to the freezing point, after which the upper cover glass was removed, leaving a solidified sulfur layer attached to the bottom plate. During many repeated experiments, we found that the index of refraction measured varied, probably dependent on the orientation of the constituent orthorhombic ( $\alpha$ ) and monoclinic ( $\beta$ ) crystals. (The real part of the refractive index of orthorhombic sulfur is known at visible wavelengths to vary by as much as 15%, depending on the orientation of these birefringent crystals. See, e.g., the *International Critical Tables*.) Inspection of the solid surface revealed, for some samples, a mosaic structure defined by polycrystalline threadlike patterns that may fill part or the whole of the surface. Other samples prepared by the same method were essentially plane, glassy-looking surfaces at the beginning (immediately after glass separation), but partially or wholly deteriorated with time after one to several hours—characteristic of the  $\beta$  to  $\alpha$  transformation. As the light spot falling on the sample is 1–2 mm in diameter, we were able to measure different regions of the same sample to look for possible correlation between the  $n$  values measured and the nature of the inspected region. All samples so prepared are referred to in the following as Type X. Type Y will refer to the orthorhombic crystalline samples which we describe next. A comparison between solid sulfur, Types X and Y, is presented below.

To obtain a well-defined form of solid sulfur, we prepared orthorhombic crystalline solid sulfur, which is the stable form at temperatures below 95°C. Unlike monoclinic sulfur, it is free of needlelike crystals. To prepare orthorhombic sulfur, the oven was kept at a constant temperature of 130°C, and two glass plates were maintained at constant temperature between 88 and 93°C. After the liquid is poured onto one of the glass plates, it is allowed for several min-

utes to supercool to the glass temperature. After the cover glass is placed on the liquid sulfur, crystallization takes place slowly; when it is completed, it is kept at that temperature for several minutes before the sandwich is removed for cooling to room temperature. An almost homogeneous glassy-looking surface is obtained which does not deteriorate with time, at least over several days.

Verification that our Type Y sulfur sample is in fact  $\alpha$  orthorhombic was obtained with a polarizing microscopy, a 50 $\times$  objective lens, a substage conoscopic lens, and crossed polaroid filters (cf. Phillips, 1971). More than 10 separate interference figures were obtained. In every case, isogyre curvature, and the optical sign (obtained with an accessory plate into which a 550-nm retardation plate was inserted) were consistent with known values for orthorhombic sulfur ( $2V = 68^\circ 58'$ , +), and inconsistent with known values for monoclinic sulfur ( $2V = 58^\circ$ , -).

#### TRANSMISSION MEASUREMENT OF LIQUID SULFUR

The transmittance of liquid sulfur at 133°C was measured, using a Cary Model 14PM spectrometer. A rectangular quartz cell was fitted to a cavity in an aluminum block to make good thermal contact with the block, which was heated by a heating element. The temperature was recorded by a thermocouple fastened to the block. Sulfur fragments were introduced into the cell and heated, and the molten sulfur then kept at a constant 133°C. The light passed through a hole drilled in the block and was then transmitted through the liquid sulfur. Here also, care was taken to remove gas bubbles adhering to the cell wall. In addition, the temperature readings were corrected by comparison with the readings of another thermocouple dipped into the middle of the cell. The temperatures cited are accurate to within  $\pm 2^\circ\text{C}$ . The samples were measured after 3 hr at constant tempera-

ture, for the same reason mentioned above for the reflection measurements.

To obtain the transmittance of liquid sulfur, we compared the measured transmission values with those for an empty cell (with the same geometric configuration) and made a correction for the reflectance at the quartz-air interface compared with that at the quartz-liquid sulfur interface. For this correction, we used the values of  $n$  which we measured by ellipsometry for liquid sulfur; the  $n$  values for quartz are known from the literature. The imaginary part,  $k$ , of the refractive index was calculated using the relation  $T_s = e^{-4\pi kd/\lambda}$ , where  $d$  is the thickness of the liquid sample (1 cm),  $\lambda$  is the wavelength, and  $T_s$  is the transmission of liquid sulfur.  $T_s = (T/T_0)(1 - r_0)^2/(1 - r)^2$ , where  $T$  and  $T_0$  are the transmitted intensities through the liquid in the cell and through the empty cell, respectively;  $r_0$  is the reflectance at the quartz-air interface and is given by  $r_0 = (n_q - 1)^2/(n_q + 1)^2$ , where  $n_q$  is the refractive index of quartz; and  $r$  is the reflectance at the quartz-liquid interface and is given by  $r = (n - n_q)^2/(n + n_q)^2$ , where  $n$  is the real part of the refractive index of liquid sulfur. The reflectance,  $R$ , of liquid sulfur was calculated according to the normal incidence Fresnel equation,  $R = (n - 1)^2/(n + 1)^2$ , where  $n$  is the measured index of liquid sulfur.

#### RESULTS AND DISCUSSION

Table I shows  $n$  and  $k$ , and the specular reflectance,  $R$ , for the surface layer of liquid sulfur at 133°C, as measured between 0.4 and 2.0  $\mu\text{m}$ . The uncertainty in  $n$  is  $\pm 0.003$  for the range 0.4–0.8  $\mu\text{m}$  and  $\pm 0.01$  for 0.9–2.0  $\mu\text{m}$ . The uncertainty in  $k$  for values higher than about  $10^{-5}$  is  $\pm 30 \times 10^{-8}$ . For lower  $k$  values, the uncertainty drops to about  $\pm 4 \times 10^{-8}$ . Although not shown in the table,  $k$  values of  $0.008 \pm 0.002$  at 0.4  $\mu\text{m}$  and  $(5.3 \pm 0.2) \times 10^{-4}$  at 0.45  $\mu\text{m}$  were derived from transmission measurements on thin liquid films, respec-

TABLE I  
OPTICAL CONSTANTS AND NORMAL INCIDENCE  
SPECULAR REFLECTANCES FOR LIQUID  
SULFUR AT 133°C

$\lambda(\mu\text{m})$	$k(\times 10^{-4})$	$n$	$R$
0.4		2.038	0.1167
0.45		1.971	0.1068
0.51	1830	1.930	0.1007
0.515	1410	1.927	0.1003
0.52	1105	1.924	0.0999
0.525	873	1.922	0.0996
0.53	646	1.920	0.0993
0.54	378	1.916	0.0987
0.55	248	1.912	0.0980
0.56	160	1.909	0.0976
0.57	101	1.906	0.0972
0.58	75	1.903	0.0968
0.59	59	1.900	0.0963
0.6	44	1.897	0.0959
0.65	25	1.886	0.0942
0.7	24	1.877	0.0929
0.75	22	1.871	0.0920
0.8	20	1.867	0.0914
0.9	18	1.858	0.0901
1.0	18	1.852	0.0892
1.2	17	1.845	0.0882
1.4	16	1.842	0.0878
1.6	15	1.840	0.0875
1.8	20	1.840	0.0875
2.0	23	1.840	0.0875

tively 15 and 50  $\mu\text{m}$  thick, at a temperature  $\approx 140^\circ\text{C}$ .

The  $n$  values for the solid, Types X and Y, were measured at one wavelength (0.6  $\mu\text{m}$ ) and confined only to freshly prepared samples, since Type X undergoes marked changes with time. Measurements were made on 20 different regions of 10 samples of Type X and on 13 different regions of 3 samples of Type Y. The results were:  $n$  (at 0.6  $\mu\text{m}$ ) =  $1.97 \pm 0.12$  for Type X and  $1.99 \pm 0.07$  for Type Y. The difference between these values is smaller than the probable error of measurement and implies negligible differences in calculated reflectances. The larger scatter and the aging factor in Type X are consequences of the undefined and unstable allotropic forms into which Type X may solidify. On the other hand, Type Y is

the more stable orthorhombic form, and the  $n$  and  $R$  values displayed in Table II are for Type Y. For that part of the visible spectrum for which measurements of  $\alpha$  sulfur are displayed in the *International Critical Tables*, our results for Type Y are consistent with orthorhombic crystals, the AB plane parallel to the glass. For randomly oriented crystals, a higher value of  $n$  would be expected. Figure 2 shows the wavelength dependence of  $n$  for solid orthorhombic sulfur (Type Y) and liquid sulfur (133°C), and of  $k$  for liquid sulfur.

As shown in Table I,  $R$  is determined by  $n$  since  $k \ll n$  in this region. It decreases with increasing wavelength throughout the entire wavelength region measured; the greater the excursion from the near-ultraviolet absorption "edge" of elemental sulfur, the lower  $R$  is. For this reason the reflectance of liquid sulfur when measured without special precautions is likely to be composed of a weak surface reflectance, together with the more dominant long-wave transmitted light, which is scattered and re-

TABLE II  
REFRACTIVE INDEX AND  
NORMAL INCIDENCE SPECULAR  
REFLECTANCES FOR SOLID  
ORTHORHOMBIC SULFUR  
AT 25°C

$\lambda(\mu\text{m})$	$n$	$R$
0.4	2.12	0.129
0.45	2.06	0.120
0.5	2.03	0.116
0.55	2.01	0.113
0.6	1.99	0.110
0.65	1.98	0.108
0.7	1.97	0.107
0.75	1.97	0.106
0.8	1.96	0.105
0.9	1.95	0.104
1.0	1.95	0.103
1.2	1.94	0.102
1.4	1.94	0.102
1.6	1.94	0.102
1.8	1.94	0.102
2.0	1.94	0.102

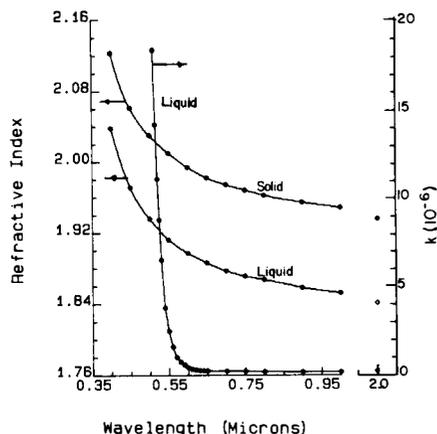


FIG. 2. The wavelength dependence of the real refractive index of sulfur (solid and liquid) and of the imaginary index,  $k$ , of liquid sulfur.

flected back from the undersurface of the liquid or from suspended particles. Thus, such a spectrum, which is also sometimes referred to as a reflectance spectrum, should also show increasing "reflectance" with increasing wavelength for liquid sulfur, as is reported (Nelson *et al.*, 1983). Because non-surface reflection is suppressed in the work reported here, we find a reflectivity about half that reported for liquid sulfur at comparable temperatures by Nelson *et al.* (1983); however, this serves to strengthen, not weaken, their conclusion that liquid sulfur melts on Io would be classified as "black" by the Voyager spectrophotometric analysis (Soderblom *et al.*, 1980).

Note that  $R$  for the solid sulfur sample is at most 2% greater than for the liquid. The variation of  $R$  with temperature,  $T$ , for  $120^{\circ}\text{C} \leq T \leq 180^{\circ}\text{C}$ , is <1% over the entire wavelength range  $0.4 \mu\text{m} \leq \lambda \leq 2.0 \mu\text{m}$  (Sasson and Arakawa, in preparation). Gradie and Veverka (1984) have found that the reflectance of powdered sulfur decreases with decreasing particle size at short visible wavelengths but increases with decreasing particle size at long visible wavelengths; their results make clear that particle size effects must be included in using optical

constants of solid sulfur, such as those reported here, in calculating reflectivities.

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