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## Infrared band strengths and other properties of amorphous and crystalline dimethyl ether

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### ABSTRACT

Laboratory astrochemists have generated infrared (IR) data for nearly all common classes of organic compounds, but ethers in the solid phase continue to be neglected despite detections of ethers in the interstellar medium by radio astronomers and uncertainty in how extraterrestrial ethers are formed. To address this paucity of data, here we present new mid-IR spectra of amorphous and crystalline dimethyl ether, (CH<sub>3</sub>)<sub>2</sub>O, the simplest member of its class. Spectral positions are tabulated and compared to previous results, but more importantly we also report IR band strengths and absorption coefficients, which we have not found in the literature and on which quantitative IR studies depend. Optical constants of amorphous and crystalline dimethyl ether also have been calculated. Some applications are described.

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### 1. Introduction

Infrared spectroscopy is still the most direct remote-sensing method for *in situ* molecular identifications on icy and rocky surfaces of astronomical objects, backed by reference spectra obtained in terrestrial laboratories. In addition, studies of laboratory analog ices continue to rely heavily on reference spectral data for probing reaction chemistry at low temperatures (<~100 K). For quantitative work, it is important that such reference spectra be characterized not only by absorbance positions (e.g., wavenumber, wavelength), but also by spectral intensities. However, it is our experience that infrared intensity measurements at relevant temperatures still have not been published for many astrophysically important solid compounds.

Of the common classes of organic molecules of astrochemical interest, there have been few, if any, reports of IR intensities of ethers in the solid state. In past publications we have reported on members of the hydrocarbon [1,2] and alcohol [3] families, an ester [4], a ketone [5], an aldehyde [6], several nitriles [7], and four thiols [8]. In the case of ethers, the only band-strength measurements we are aware of are those we reported for propylene oxide, (c-OC<sub>2</sub>H<sub>3</sub>)CH<sub>3</sub> [9]. In this brief paper, we report on the first such measurements for the prototypical dimethyl ether, (CH<sub>3</sub>)<sub>2</sub>O.

The first identification of interstellar (CH<sub>3</sub>)<sub>2</sub>O was made by radio astronomers over 40 years ago, and since then dimethyl ether has been found in a variety of interstellar sources [10,11]. However, the origin of (CH<sub>3</sub>)<sub>2</sub>O remains a subject of debate, with some arguing for a gas-phase path [12] and others favoring formation on the surfaces of ice-coated interstellar grains [13]. Investigations of the latter often involve using IR spectroscopy to study icy solids, and in such cases laboratory measurements of reference spectra are invaluable. An important application is the use of such spectra to identify reaction products generated by photochemical or radiolytic methods. Another application is the use of band strengths to determine reaction yields or the amounts of a starting material present. In making multi-component ice mixtures, it is important to know the amount of each component initially present, and this can be done by careful calibration using the IR band strengths of each individual substance, but only if such band strengths are already known.

To date, no computational method has been able to produce IR band strengths or absorption coefficients of ices that reliably match those delivered by laboratory measurements. Until such a method is developed, astrochemists must continue to rely on laboratory results of IR intensities, whether the application is to terrestrial studies or astronomical observations of extraterrestrial objects and IR spectra.

Motivated by these considerations, here we present new results on solid dimethyl ether obtained in our laboratory. We report density and refractive index data that then are combined with new spectral

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measurements to yield IR absorption coefficients and band strengths for amorphous and crystalline dimethyl ether. Optical constants also have been calculated.

## 2. Experimental

The equipment and procedures employed are described in our recent papers, so only a summary is needed [14,15]. Dimethyl ether (99+% purity) was purchased from Sigma Aldrich and used without additional purification.

Infrared spectra were obtained with a Thermo iS50 spectrometer at  $1\text{-cm}^{-1}$  resolution over  $6000$  to  $450\text{ cm}^{-1}$ , with 100 to 200 scans per spectrum. Dimethyl ether was deposited onto a pre-cooled CsI substrate to a thickness of about  $0.5$  to  $4\text{ }\mu\text{m}$ , at a rate of a few micrometers per hour as measured by laser interferometry at  $670\text{ nm}$ . The vacuum system's background pressure was  $\sim 10^{-8}$  Torr, and the deposition temperatures were  $10$  and  $75\text{ K}$ , producing amorphous and crystalline ices, respectively. The unpolarized IR beam was aligned perpendicular to the plane of the ice sample. Errors and uncertainties for band strengths ( $A'$ ) and absorption coefficients ( $\alpha'$ ) are discussed in Hudson et al. [14] and are on the order of 5%, and less for the stronger IR features.

For details about the ultra-high vacuum ( $\sim 10^{-10}$  Torr) system used to measure ice densities and refractive indices, again see recent papers from our laboratory [16]. Briefly, refractive indices were determined by two-laser interferometry, again at  $670\text{ nm}$ , and densities were determined with a quartz crystal microbalance. See earlier papers for more information on the use of a quartz crystal microbalance [17], two-laser interferometry for measuring refractive indices [18], and determinations of ice thickness with a single laser [19].

## 3. Results

### 3.1. Refractive indices and densities

Triplicate measurements gave refractive indices of  $n(15\text{ K}) = 1.33$  and  $n(75\text{ K}) = 1.44$  for amorphous and crystalline dimethyl ether, respectively. A density of  $\rho(15\text{ K}) = 0.874\text{ g cm}^{-3}$ , was measured for amorphous dimethyl ether, and  $\rho(75\text{ K}) = 0.970\text{ g cm}^{-3}$  for crystalline  $(\text{CH}_3)_2\text{O}$ . The only  $n$  or  $\rho$  found in the literature was  $\rho(93\text{ K}) = 0.975\text{ g cm}^{-3}$  from a crystallographic study [20], which agreed with our work. Uncertainties were on the order of  $\pm 0.005$  for  $n$  and  $\pm 0.005\text{ g cm}^{-3}$  for  $\rho$ . Again, see our earlier papers for details on the equipment and methods employed.

### 3.2. Infrared spectra

Fig. 1 shows mid-IR spectra of  $(\text{CH}_3)_2\text{O}$  deposited at  $10\text{ K}$  to give the amorphous form of the compound and at  $75\text{ K}$  to give the crystalline phase. The crystalline ice also could be obtained in a few minutes by warming the amorphous solid to about  $65\text{ K}$ , and the change was irreversible. Continued warming to about  $110\text{ K}$  gave complete sublimation of the sample in our vacuum system in only a few minutes. Fig. 2 shows expansions of two IR regions. No crystalline-crystalline phase changes were observed, and none were expected based on earlier calorimetric work [21].

Our IR spectra closely resemble the few that are in the literature, such as those of Shriver-Mazzuoli et al. for amorphous  $(\text{CH}_3)_2\text{O}$ , recognizing that their spectra were obtained by reflection from samples deposited onto a gold surface [22]. Our spectra also match those of Allan et al. for crystalline  $(\text{CH}_3)_2\text{O}$  [23]. Peak positions agree well with these earlier papers. All of the published spectra of solid dimethyl ether we have located to date are missing regions covered in our figures or they are for a reflection mode, hindering a more-detailed comparison.

The motivation for our study was not a full vibrational analysis, as it is doubtful that we could improve on existing work in that area. We also did not aim for an assignment of each IR peak seen or any sort of

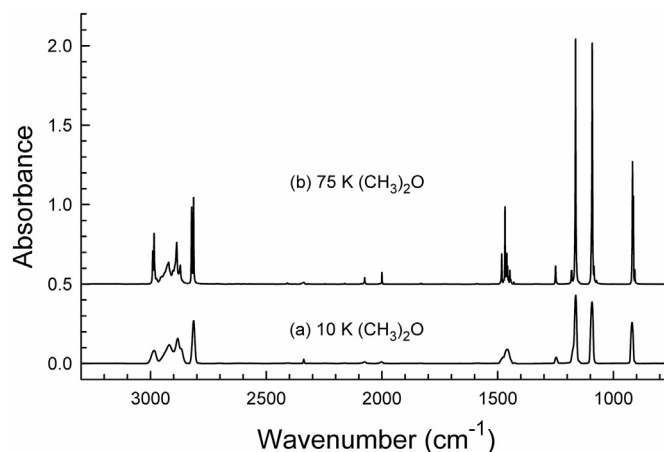


Fig. 1. Survey spectra of (a) amorphous dimethyl ether deposited at  $10\text{ K}$  and (b) crystalline dimethyl ether deposited at  $75\text{ K}$ . The original thickness of each ice was about  $1\text{ }\mu\text{m}$ . Spectra have been offset for clarity.

reanalysis. Instead, our investigation was driven by the need for measurements of spectral intensity required for astrochemical work. Accordingly, we selected nine IR regions over our mid-IR range in which to measure apparent absorption coefficients, denoted  $\alpha'$ , of IR peaks and to integrate over each region to determine apparent band strengths, denoted  $A'$ . To obtain both  $\alpha'$  and  $A'$ , six ices were prepared with thicknesses of about  $0.5$  to  $2\text{ }\mu\text{m}$ , their IR spectra were recorded, peak heights and band areas were measured, and  $\alpha'$  and  $A'$  were obtained from the slopes of standard Beer's Law plots. See Hollenberg and Dows for the original description of the method used [24], and our earlier papers for other examples (e.g., [1,3,5]). All such Beer's Law graphs had correlation coefficients of  $0.998$  and higher.

Table 1 lists the peaks and regions studied for amorphous  $(\text{CH}_3)_2\text{O}$ , and Table 2 does the same for the crystalline phase. Descriptions are given for the vibrations in each region, but we caution that these descriptions are highly simplified. See Levin et al. for more details [25]. Note that a few regions are sufficiently complex that one finds different assignments in the literature (e.g.,  $2970\text{--}2840$ ,  $1550\text{--}1400\text{ cm}^{-1}$ ).

### 3.3. Optical constants

We also have used our spectra to calculate the mid-IR real ( $n$ ) and imaginary ( $k$ ) components of the complex index of refraction of amorphous and crystalline  $(\text{CH}_3)_2\text{O}$ . As in our earlier papers, this was done

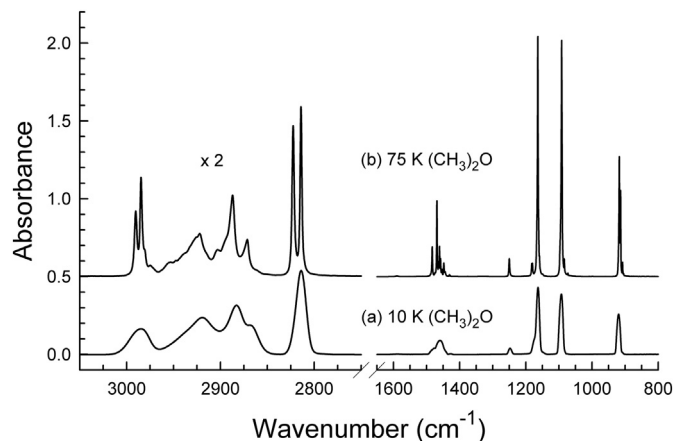


Fig. 2. Expansions of two regions from Fig. 1. (a) amorphous dimethyl ether deposited at  $10\text{ K}$  and (b) crystalline dimethyl ether deposited at  $75\text{ K}$ . The original thickness of each ice was about  $1\text{ }\mu\text{m}$ . Spectra have been offset for clarity. Note the vertical expansion by a factor of 2 on the left.

**Table 1**  
Selected infrared absorptions of amorphous dimethyl ether at 10 K.

Peak position (cm <sup>-1</sup> )	$\alpha'$ (cm <sup>-1</sup> ) <sup>a</sup>	Integration range (cm <sup>-1</sup> )	$A'$ (10 <sup>-18</sup> cm molec <sup>-1</sup> ) <sup>b</sup>	Approximate description <sup>c</sup>
2985	1870	3025–2840	21.64	CH <sub>3</sub> stretches + overtone, comb.
2814	5790	2840–2790	6.81	CH <sub>3</sub> stretches
2075	222	2120–2040	0.37	Overtone <sup>d</sup>
2002	221	2030–1975	0.30	Overtone <sup>d</sup>
1459	1890	1510–1410	4.84	CH <sub>3</sub> def.
1248	775	1266–1220	0.78	CH <sub>3</sub> rock, def.
1163	9810	1190–1135	10.67	CH <sub>3</sub> rock, def.
1093	8680	1110–1070	8.99	C-O asymm. stretch
920	5720	935–900	5.58	C-O symm. stretch

<sup>a</sup> From the slopes of Beer's Law graphs of  $2.303 \times$  (peak height) against ice thickness. In each case, the slope is the apparent absorption coefficient,  $\alpha'$ .

<sup>b</sup> From the slopes of Beer's Law graphs of  $2.303 \times$  (band area) against ice thickness. In each case, the slope divided by  $(\rho N_A/M)$  gives  $A'$ , where  $M$  = molar mass = 46.07 g mol<sup>-1</sup>,  $N_A$  =  $6.022 \times 10^{23}$  molecules mol<sup>-1</sup>, and density = 0.874 g cm<sup>-3</sup>. For recent examples, see Gerakines and Hudson (2015).

<sup>c</sup> See Levin et al. (1978) for more-complete descriptions.

<sup>d</sup> Peaks near 2075 and 2002 cm<sup>-1</sup> are assigned to  $\nu_{20} + \nu_6$  and  $\nu_{17} + \nu_6$  overtones, respectively.

using an iterative Kramers-Kronig method [7]. Figs. 3 and 4 shows the results for amorphous and crystalline dimethyl ether, respectively, and our home page has electronic files with optical constants  $n$  and  $k$  (<https://science.gsfc.nasa.gov/691/cosmicice>). One advantage of optical constants over band strengths is that  $n$  and  $k$  can be used to calculate IR transmission spectra as well as IR spectra of samples deposited onto a metal substrate for observation by specular reflection [26,27]. As in earlier papers, we have adhered to the Nebraska Convention in our choice of signs for the complex index of refraction,  $n - ik$  [28].

## 4. Discussion

### 4.1. Infrared spectra

As already stated, our IR spectra of amorphous and crystalline (CH<sub>3</sub>)<sub>2</sub>O agree qualitatively with the few spectra that have been published. However, quantitative comparisons to the intensities in our Tables 1 and 2 are difficult since earlier publications lack either a numerical scale on the vertical axes of spectra or direct measurements of ice thickness. Aside from previous work, our results also agree with expectations based on our experience with a variety of organic ices, as well as those of other workers going back many years [29]. Deposition at a sufficiently low temperature produces a glassy, amorphous solid, with broad IR peaks displaying little or no structure. Warming irreversibly initiates crystallization accompanied by splitting, shifts, and sharpening of IR peaks, differences that also are seen on comparing spectra of ices prepared at 10 and 75 K, traces (a) and (b) in Figs. 1 and 2.

The sharpness of the IR peaks of crystalline (CH<sub>3</sub>)<sub>2</sub>O is reflected in the fact that the average value of  $\alpha'$  in Table 2 is about 300% (factor of 4) larger than the average value of  $\alpha'$  in Table 1 for amorphous dimethyl

ether. In contrast, the average value of  $A'$  for the crystalline ice, reflecting IR band areas, is only about 10% larger than that of the amorphous solid. Put another way, the band areas of the amorphous and crystalline ices are similar, but the peak heights and band widths are not.

The entries of Tables 1 and 2 include most of the stronger absorptions of (CH<sub>3</sub>)<sub>2</sub>O, but many of the regions listed cover multiple vibrational modes that are hard to unravel, measure, and assign in solid-phase spectra, particularly in the amorphous ice with its broad IR bands. Three exceptions are the strong features at 1163 ( $\nu_{20}$ ), 1093 ( $\nu_{17}$ ), and 920 ( $\nu_6$ ) cm<sup>-1</sup> for amorphous (CH<sub>3</sub>)<sub>2</sub>O. Barely discernable in Fig. 1 are combination bands at 2075 ( $\nu_{20} + \nu_6$ ) and 2002 ( $\nu_{17} + \nu_6$ ) cm<sup>-1</sup> that also are found in earlier papers [e.g., 22]. Peaks from <sup>13</sup>C and <sup>18</sup>O isotopologs were observed at 1159 (<sup>13</sup>C,  $\nu_{20}$ ), 1084 (<sup>13</sup>C,  $\nu_{17}$ ), 1073 (<sup>18</sup>O,  $\nu_{17}$ ), 908 (<sup>13</sup>C,  $\nu_6$ ), and 905 (<sup>18</sup>O,  $\nu_6$ ) cm<sup>-1</sup>, in agreement with previous work [23,25]. None of these features were studied in any detail, but their identifications helped to confirm the purity of our samples by showing that such small peaks are not due to contaminants. We note here that the numbering we have used for vibrational modes is that of McKean et al. [30] and Ilieva et al. [31] and differs from that used by Levin et al., who reversed the  $b_1$  and  $b_2$  modes [25]. See also the tabulations of Kanazawa and Nukada [32].

Several sets of gas-phase IR intensities are available for dimethyl ether, but for the solid compound we know of only one, consisting of three  $A'$  values and the relative intensities of four IR bands, with the  $A'$  values being based on an indirect measurement on a gas-phase mixture [33]. The resulting ratios (relative intensities) of the 1248 ( $\nu_5$ ), 1163 ( $\nu_{20}$ ), 1093 ( $\nu_{17}$ ), and 920 ( $\nu_6$ ) cm<sup>-1</sup> bands of amorphous (CH<sub>3</sub>)<sub>2</sub>O at 15 K are 0.06:1:0.83:0.49, close to our own result of 0.07:1:0.84:0.52.

For the  $A'$  values of dimethyl ether's IR bands, Scheltinga et al. [33] published three values based on (i) reference measurements with a

**Table 2**  
Selected infrared absorptions of crystalline dimethyl ether at 75 K.

Peak position (cm <sup>-1</sup> )	$\alpha'$ (cm <sup>-1</sup> ) <sup>a</sup>	Integration range (cm <sup>-1</sup> )	$A'$ (10 <sup>-18</sup> cm molec <sup>-1</sup> ) <sup>b</sup>	Approximate description <sup>c</sup>
2984.7	7130	3030–2840	19.69	CH <sub>3</sub> stretches + overtone, comb.
2814.1	13300	2840–2750	5.96	CH <sub>3</sub> stretches
2074.7	991	2100–2050	0.36	Overtone <sup>d</sup>
2000.3	1750	2030–1980	0.39	Overtone <sup>d</sup>
1468.4	11600	1500–1420	5.89	CH <sub>3</sub> def.
1250.0	2600	1260–1230	0.80	CH <sub>3</sub> rock, def.
1163.6	35100	1190–1130	11.78	CH <sub>3</sub> rock, def.
1091.7	37500	1110–1070	11.81	C-O asymm. stretch
917.4	18300	930–900	6.59	C-O symm. stretch

<sup>a</sup> From the slopes of Beer's Law graphs of  $2.303 \times$  (peak height) against ice thickness. In each case, the slope is the apparent absorption coefficient,  $\alpha'$ .

<sup>b</sup> From the slopes of Beer's Law graphs of  $2.303 \times$  (band area) against ice thickness. In each case, the slope divided by  $(\rho N_A/M)$  gives  $A'$ , where  $M$  = molar mass = 46.07 g mol<sup>-1</sup>,  $N_A$  =  $6.022 \times 10^{23}$  molecules mol<sup>-1</sup>, and density = 0.970 g cm<sup>-3</sup>. For recent examples see Gerakines and Hudson (2015).

<sup>c</sup> See Levin et al. (1978) for more-complete descriptions.

<sup>d</sup> Peaks near 2075 and 2000 cm<sup>-1</sup> are assigned to  $\nu_{20} + \nu_6$  and  $\nu_{17} + \nu_6$  overtones, respectively.

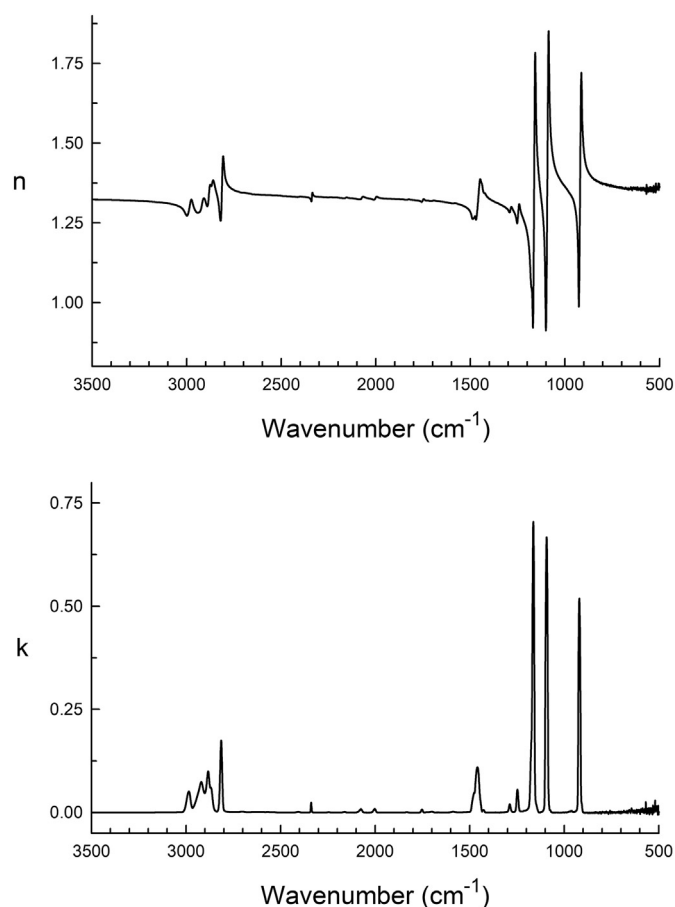


Fig. 3. Optical constants of amorphous dimethyl ether deposited at 10 K.

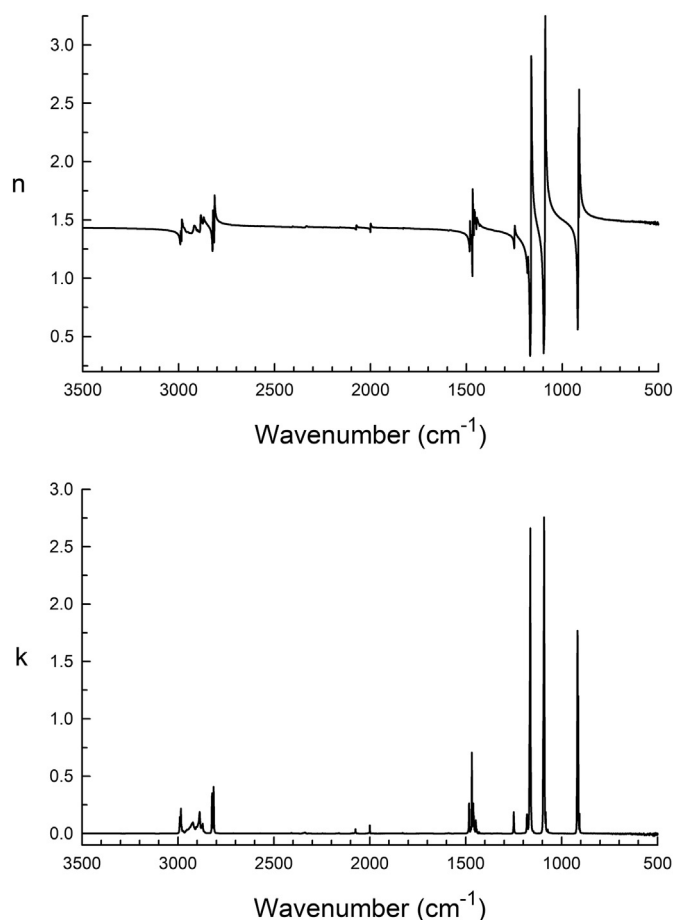


Fig. 4. Optical constants of crystalline dimethyl ether deposited at 75 K.

gas-phase 1:1  $\text{CH}_4 + (\text{CH}_3)_2\text{O}$  mixture, (ii) an assumption about the composition of an ice made from this gas-phase mixture, and (iii) a literature value for a band strength of pure  $\text{CH}_4$ . Although the resulting  $A'$  values differed by no more than  $>10\%$  from those reported here, the validity of any such quantitative comparison is unknown as no integration limits were provided for the three IR bands measured by previous workers. Regardless, the new results presented in the present paper permit better determinations of ice composition in dimethyl ether mixtures through independent calibrations of the ether concentration in an ice, through separate deposition lines, removing the need to rely on data for a gas-phase mixture.

#### 4.2. Some applications

The challenges in observing and identifying IR features of dimethyl ether in an interstellar ice were discussed some years ago by Peeters et al. [34], and we agree with their conclusions about the difficulties involved. The overlap of all  $(\text{CH}_3)_2\text{O}$  infrared features with those of abundant ice and grain components is sufficiently great as to be at the limit, if not beyond, current technology. The high sensitivity of the James Webb Space Telescope (JWST), scheduled for launch in 2021, may offer new and better possibilities for  $(\text{CH}_3)_2\text{O}$  detection in interstellar ices.

We suspect that the primary use of our data will be in the work of laboratory astrochemists investigating the chemical and physical properties of dimethyl ether. For example, Schriver et al. have explored the photochemical behavior of  $\text{H}_2\text{O} + (\text{CH}_3)_2\text{O}$  (5:1) ices at 10 K [35], and have described an intramolecular decomposition to account for the products, while Peeters et al. studied the photostability of ices containing  $(\text{CH}_3)_2\text{O}$  [34]. Both types of experiments can be better quantified by using our new data, in one case to better determine the initial ice composition and in the other to quantify the number of  $(\text{CH}_3)_2\text{O}$  molecules

destroyed by a given photolytic exposure. Conversely, our solid-phase band strengths and optical constants now make it possible to determine  $(\text{CH}_3)_2\text{O}$  yields in reactions induced by the radiolysis or photolysis of  $\text{CH}_3\text{OH}$  ices. See the paper of Sullivan et al. and references therein [36]. Our band strengths and other data also can be used to determine vapor pressures of solid dimethyl ether using the method of Khanna et al. [37]. Finally, since dimethyl ether in astronomical solids almost certainly will exist in the presence of ices such as  $\text{H}_2\text{O}$  and  $\text{CO}_2$ , our results can be used to better determine ether compositions in ices consisting of more than one component, such as through the use of multiple deposition lines.

#### 5. Summary and conclusions

Here we have reported the first direct measurements of IR band strengths and absorption coefficients for amorphous and crystalline  $(\text{CH}_3)_2\text{O}$ . Refractive indices, densities, and optical constants also have been determined, and the latter made available in electronic form. We anticipate that these results will be of use in the continuing effort to determine the source and evolution of extraterrestrial dimethyl ether, such as its formation by photo- and radiation-chemicals paths, and to measurements of the compound's vapor pressures.

#### CRediT authorship contribution statement

**Reggie L. Hudson:** Conceptualization, Methodology, Writing - original draft. **Yukiko Y. Yarnall:** Investigation, Formal analysis, Writing - original draft. **Falvia M. Coleman:** Investigation, Formal analysis, Writing - original draft.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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