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Infrared Intensities and Molar Refraction of Amorphous Dimethyl Carbonate – Comparisons to Four Interstellar Molecules

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Abstract

The first measurements of infrared (IR) band intensities of solid dimethyl carbonate are presented, along with measurements of this compound's refractive index and density near 15 K, neither of which has been reported. Molar refractions are used to compare these results to other new data from ices made of methyl acetate, acetone, acetic acid, and acetaldehyde, four molecules known to exist in the interstellar medium. Comparisons are made to IR intensities taken from the literature on amorphous ices. The value and importance of comparisons based on molecular structures, to predict and test laboratory results, is highlighted.

1. Introduction

Dimethyl carbonate (DMC) possesses structural similarities to several extraterrestrial C-H-O-containing organic compounds that have been discovered in the interstellar medium (ISM), but DMC has yet to be identified there.[1] Shown in Fig. 1 are, from left to right, simplified drawings of four known interstellar molecules, an aldehyde, acid, ketone, and ester, followed by DMC. The low temperature (~10 K) of dense molecular clouds suggest that if these molecules are present in the solid state in the ISM then they probably will be in an amorphous ice, perhaps embedded in solid H_2O. With twelve atoms, DMC is sufficiently complex that its formation in a low-temperature, low-vacuum environment almost certainly would have to occur in the icy mantles of interstellar grains, and not in the gas phase.

The laboratory study of many such candidate interstellar molecules in solid form remains hindered by the lack of fundamental laboratory data, particularly low-temperature spectroscopic measurements. For icy solids, this usually means infrared (IR) spectra of such molecules in an amorphous state. With the proper spectroscopic data, including both IR positions and intensities, it is possible to devise laboratory experiments to investigate the physical state of interstellar molecules, to identify pathways to a molecule's solid-phase formation and destruction, and to make quantitative measurements of abundances, reaction yields, branching ratios, and destruction rates.

In this paper we report the first measurements of IR band intensities of solid dimethyl carbonate. We also present measurements of two physical properties needed to quantify our work, the index of refraction (n) and density (ρ) of amorphous DMC. Neither has been

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reported for this compound in a solid state, but both are needed for lab-to-lab comparisons and for the study of more-complex DMC-containing ices. Since n and ρ pairs have been published for only a few interstellar organic compounds, we also report the first n and ρ data for three known interstellar organics, chosen specifically for comparison to our new results on DMC. Molar refractions are used to examine the degree of correlation between data on room-temperature liquids and low-temperature amorphous ices.

Before proceeding, we recognize that our use of refractions for condensed-phase studies of organic compounds is far from original. As early as 1869, John Hall Gladstone, a future President of the Chemical Society, presented data related to densities and refractive indices, with attention to details of molecular structure and bonding that eventually led to extensive tables of molecular data for the prediction of physical properties.[2,3,4,5] We are unaware, however, of any such applications in the intervening 150 years to our own field of low-temperature astrochemistry.

2. Experimental

Only a brief summary of our methods is given here as they have been described in recent papers from our group.[6,7,8,9] Chemical reagents were purchased from Sigma Aldrich and used as received, except for routine freeze-pump-thaw degassing with liquid nitrogen. Spectra were recorded with a Thermo iS50 infrared spectrometer from 7000 to 400 cm^{-1}, but the lack of strong IR features at the higher wavenumbers and instrument noise at the lower meant that focused our attention on the 3500 – 500 cm^{-1} region, with spectra typically consisting of 100 or 200 scans at 1-cm^{-1} resolution. Vapor-phase depositions of each compound studied were made onto a pre-cooled KBr substrate with a disperser at the end of the deposition line to ensure a uniform ice thickness over the area sampled, and with a deposition rate that gave an increase in ice thickness of about 2 μm hr^{-1}. Ice thicknesses were measured by laser interferometry (λ = 670 nm) and were 0.5 to 4 μm. The deposition temperature for each sample was 10 – 15 K, but for simplicity will be stated as 15 K throughout this paper. An ultra-high vacuum (~10^{-10} torr) system was used to measure ice densities and refractive indices (again λ = 670 nm), the density determinations with a quartz crystal microbalance and the refractive indices with a two-laser interferometry arrangement. Again, see earlier papers from our laboratory, and references therein, for details.

3. Results

3.1 Densities and refractive indices

Our main goal was to measure IR band strengths for amorphous dimethyl carbonate, but to reach that goal it was necessary, as explained in the next section, to determine a reference refractive index and a density for our ices. See Table 1 for results for DMC, accompanied by values for four other compounds we examined, each quantity being an average of at least three measurements. These are the same compounds of Fig. 1. Table 2 lists data for the corresponding liquids[10], to which we return in our Discussion. We estimate that the standard error in our solid-phase n values is less than 0.01, probably ± 0.005, so that accuracy to two decimal places seems secure. Errors in density are on the order of ± 0.005 g cm^{-3}.

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3.2 Infrared spectra

Each ice sample's thickness (h) was monitored during the sample's growth by recording interference fringes generated by a laser's light incident on the KBr substrate, with equation (1) giving h.[11,12]

$$h = \frac{N_{fr}\lambda}{2\sqrt{n^2 - \sin^2\theta}} \tag{1}$$

In this equation, N_{fr} was the number of fringes recorded during deposition, λ = 670 nm, n was the ice's refractive index, which we determined as already described, and θ was 3.57 ± 0.04°, the angle between the incident laser beam and a line drawn perpendicular to the KBr substrate.

Previous work[13] showed that room-temperature DMC consists mainly of two molecular forms, which are drawn in Fig. 2. Of the two, the *cis-cis* conformation is the more stable, with the *cis-trans* isomer making up only ~1% of the total. With one exception, we found no evidence for the *cis-trans* isomer in our samples, and so it was not studied in the present work. In previous spectroscopic investigations of this type, it was either not observed or was too weak to identify with confidence. The only evidence we had of the *cis-trans* isomer was a very weak, noisy band near 580 cm^{-1}, from which we estimated that not more that about 1% of the sample was the less-stable conformer, as expected. For structural details of DMC, see the electron diffraction work of Mijlhoff[14], the microwave results of Lovas et al.[15], and the computational work of Kar et al.[16] or Boussessi and co-workers.[17]

Fig. 2 shows a mid-IR survey spectrum of DMC at 15 K. On warming the ice to ~110 K we observed that the IR peaks began to increase in intensity, sharpen, split, and, in some cases, show small shifts in position. All of these observations suggest that the original sample was an amorphous solid that crystallized on warming. Continued warming to 160 K resulted in our DMC ices' sublimation in our vacuum system. Fig. 4 – 6 are enlargements of selected regions of the IR spectrum of DMC after deposition at 15 K, after warming to 150 K to crystallize, and then on recooling to 15 K, showing that the changes seen on warming were irreversible.

Infrared absorption coefficients (α') for peaks and integrated intensities (A') for bands were obtained from conventional Beer's law plots. Graphs of peak height as a function of ice thickness were linear with slopes equal to (α' / 2.303), from which α' was found. Similarly, graphing band areas as a function of ice thickness gave points that could be fit with a straight line and interpreted with equation (2) for a slope equal to ($\rho_N A'$ / 2.303) from which A' was calculated.[18]

$$\int_{band} (Absorbance) d\tilde{\upsilon} = \left(\frac{\rho_N A'}{2.303}\right) h \tag{2}$$

In equation (2), ρ_N is the ice's number density found from $\rho_N = \rho\,(N_A / M)$ where N_A is Avogadro's constant, M is the molar mass (g mol^{-1}) of the compound, and ρ is the density we measured (Table 1) for DMC. Correlation coefficients of 0.996 and higher were obtained for all graphs. Table 3 summarizes our results for nine IR peaks and spectral regions. Qualitative descriptions of the vibrations for each region of interest are from Bohets and van der Veken.[13] From the α' and A' values of Table 3, it is possible to calculate similar quantities for other IR features, such as weak mid-IR bands and peaks in the near- and far-IR regions, by forming the appropriate ratios.

4. DISCUSSION

4.1 Densities and refractive indices

As just described, infrared band intensities (A') of ices rely on n and ρ, as shown by equations (1) and (2). Since few measurements of either n or ρ for ices are in the literature, it is not unusual to see liquid-phase results used in calculations involving low-temperature solids. One strategy is to use some form of the Lorentz-Lorenz equation, calculate a specific or molar refraction (R_M) from liquid-phase data, and then use the result to determine an ice's density from a measured low-temperature refractive index.[19,20,21] To examine the accuracy of such an approach, we used data from Tables 1 and 2 to calculate densities from molar refractions, found with n at 15 K and equation (3).

$$R_M = \left(\frac{M}{\rho}\right)\left(\frac{n^2-1}{n^2+2}\right) \tag{3}$$

Table 4 compares the resulting calculated densities to our measured values, with the last line showing that errors are no greater than 10% and in some cases much lower. We suspect that an upper error limit of 10% applies to similarly calculated densities for many other organic ices.

Fig. 7 shows two other way to examine our n and ρ values. The left-hand plot compares solid- and liquid-phase R_M results. Both the slope and the correlation coefficient are near one, suggesting a strong correlation, but the fact that the intercept is greater than zero again shows that caution is needed when using room-temperature liquid-phase data to calculate a density for an amorphous ice near 15 K.[22,23]

Still another, and related, way to examine our n and ρ results is by comparing our molar refractions with R_M values calculated with a simple bond-additivity approach. Table 4 gives the bond contributions published by Denbigh[24], but other sets of values[25] and more sophisticated approaches[26,27] could be used. – Taking acetaldehyde and its six bonds as an example, Table 4 leads to $R_M = 4\ (1.69) + (1.25) + (3.38) = 11.39$ cm^3 mole^{-1}. – The right-hand plot of Fig. 7 compares our measured and calculated R_M values, and again there is a high correlation coefficient (0.994), and with a much smaller y intercept than in the left-hand plot.

Here we note that the spacing of the points in the right-hand plot of Fig. 7 reflects the roughly additive nature of molar refractions.[28,29] On moving diagonally from the lower left to the upper right of Fig. 7, the separation of the first two points represents the addition of an oxygen atom to the acetaldehyde structure and the O atom's contribution to R_M. The next point is for acetone, and its greater distance from the aldehyde point is from the

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contribution of a methylene group (CH_2) to R_M. See again the structures of Fig. 1. The next two points, in the upper right, reflect the change in acetone's R_M caused by the addition of an O atom to give methyl acetate, followed by the addition of a second O atom to reach dimethyl carbonate. With additional such measurements on many compounds it might be possible to assemble a reliable set of contributions for atomic and molecular groups to be used in estimating R_M in amorphous ices. Given the time and expense of making n and ρ measurements, such R_M estimates could be valuable for checking laboratory results and for estimating ice densities when only a refractive index is available, or *vice versa*.

4.2 Infrared spectra

Dimethyl carbonate has a total of 3N – 6 = 3(12) – 6 = 30 fundamental vibrations, but many are so close or weak, or so low in frequency, that they cannot be seen in our spectra, and have not been resolved in other work on condensed-phase DMC. For example, six C-H vibrations are expected, but their overlapping positions only allow three IR features to be observed, each being assigned to two vibrational modes. These three features can just be seen in our Fig. 4a, with careful inspection of the band near 3050 cm^{-1} revealing two components and the sharper peak near 2964 cm^{-1} being the third C-H feature. In other cases, the spectral substructure reported for a single mode by previous workers[13] also appears in our data. For example, the feature near 971 cm^{-1} is reported to have a shoulder about 11 cm^{-1} lower. Again, careful inspection of our results, specifically Fig. 6a, shows both the peak and the shoulder. In short, the mid-IR spectra of our amorphous DMC has all of the same peaks and smaller features previously reported. Little attention was paid to crystalline DMC in our work, but our 150-K spectra also compare favorably with published results, despite some differences in ice preparation and temperature.[30,31]

Comparisons of the IR band strengths in our Table 3 to results from earlier papers proved to be more difficult than comparisons of peak positions. Quantitative comparisons proved to be impossible because previous publications on solid DMC did not include both an ice thickness and IR spectra with numbers along the vertical axis. However, on a qualitative level our IR intensities in Table 3 match the descriptions of some earlier workers. Certainly our results are in qualitative agreement with the most complete study of the IR spectra of solid DMC we have found, the work of Bohets and van der Veken published in this journal.[13]

One of the original motivations for this study was our desire to measure IR intensities in amorphous ices at a temperature relevant to the ISM. Unfortunately, little band-strength data is available for comparison to our DMC results. Table 6 lists three IR band strengths from our own laboratory and one from elsewhere, each compound having a carbonyl group and only C, H, and O atoms. It is seen that our dimethyl carbonate measurement for A' (C=O) is of the same magnitude as the other three intensities listed. Also, the A' (C=O) values of methyl formate and methyl propionate are sufficiently close that we feel confident in predicting that A' (C=O) will be similar for the intermediate ester, methyl acetate. Additional measurements of other aldehyde, ketone, ester, and acid ices are needed for comparison to the four entries of Table 6.

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4.3 Final comments on applications

The spectral overlap of most of the larger IR bands of DMC with those of other molecules means that using our data to identify DMC in IR spectra of interstellar ices will be challenging. It is more likely that our spectra and band intensities will find applications in laboratory studies of solid DMC's formation and reaction chemistry. Our results also will be useful in the continued effort to measure band strengths of small molecules, providing data on a relatively inexpensive, low toxicity material that can help facilitate comparisons between laboratories.

Finally, we note that nearly all publications reporting A', n, and ρ values of astrochemical ices have emphasized the influences of ice temperature and composition.[32,33] Put another way, the role of chemical structure and comparisons based on functional groups has suffered near total neglect. The result has been a tendency to treat each molecule in isolation with little or no consideration for possible trends in either a homologous or isoelectronic series or among various functional groups, such as the set in Fig. 1. Measurements and comparisons among related molecules could be a valuable way to uncover patterns, check results, and expose suspect measurements.

5. SUMMARY

Mid-IR transmission spectra of amorphous dimethyl carbonate have been measured and IR intensities calculated for the first time. New data have been presented for densities and refractive indices of solid dimethyl carbonate and ices made from four other compounds. Comparisons are made (1) between the molar refractions of solid- and liquid-phase samples and (2) between measured molar refractions of five ices and the R_M values calculated by treating molar refraction as an additive property. Linear relations are found in both cases. The use of molecular structural details in the study of amorphous-ice properties is highlighted.

ACKNOWLEDGEMENTS

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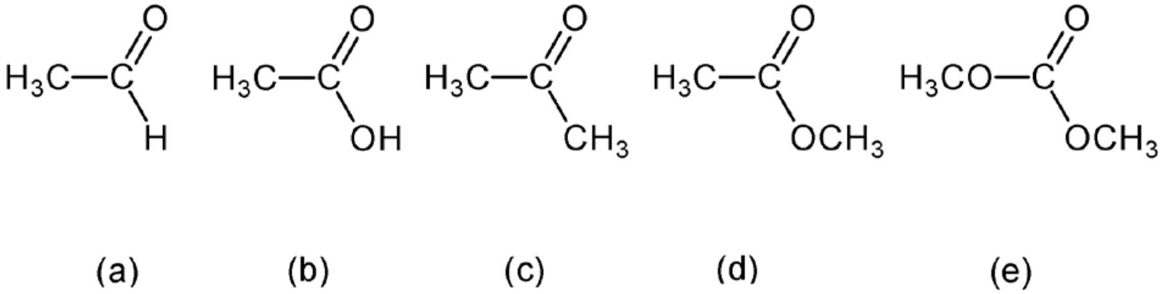

Fig. 1.

Five carbonyl-containing molecules, (a) acetaldehyde, (b) acetic acid, (c) acetone, (d) methyl acetate, and (e) dimethyl carbonate. Molecules (a) - (d) have been identified in the interstellar medium.

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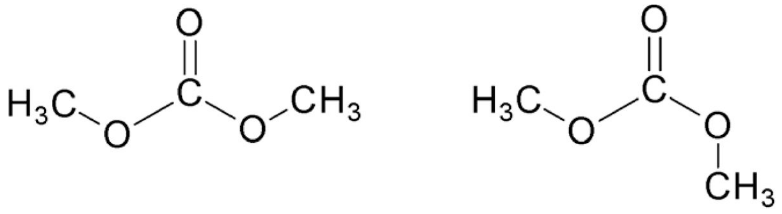

(a) cis-cis (b) cis-trans

Fig. 2.

Two conformational isomers of dimethyl carbonate.

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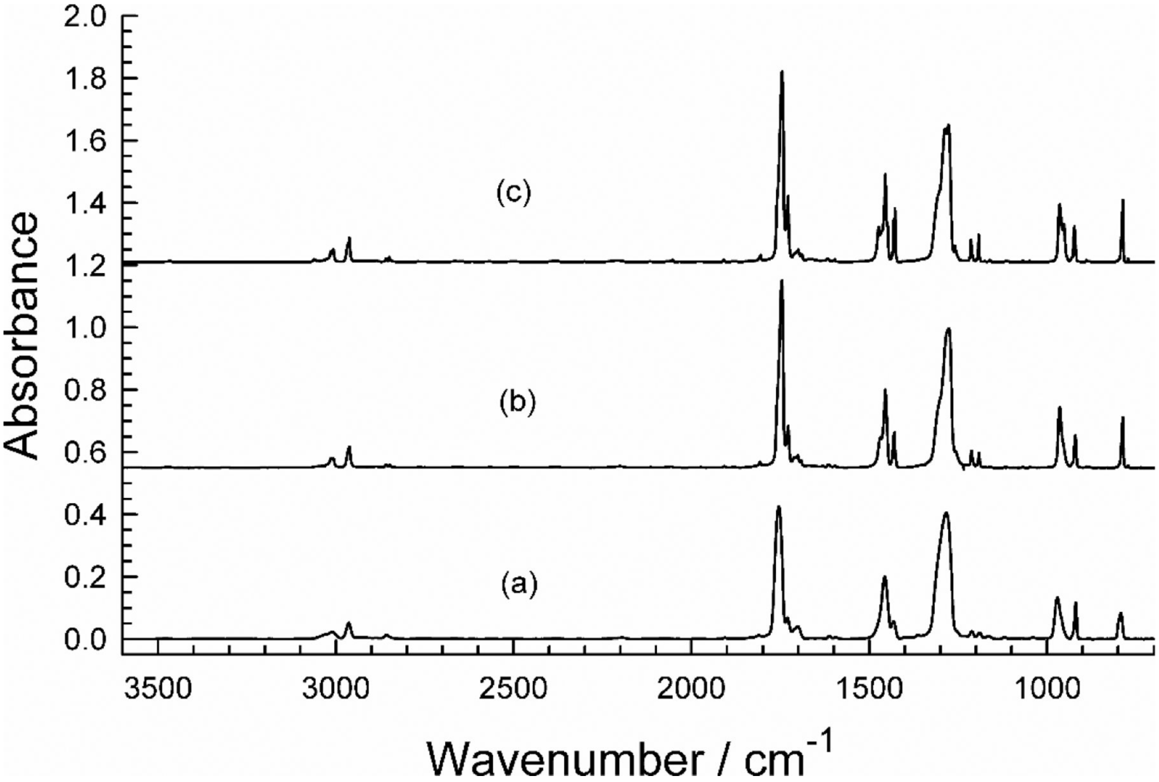

Fig. 3.

Survey spectra of dimethyl carbonate (a) deposited at 15 K, (b) warmed to 150 K, and (c) recooled to 15 K. Spectra have been offset for clarity.

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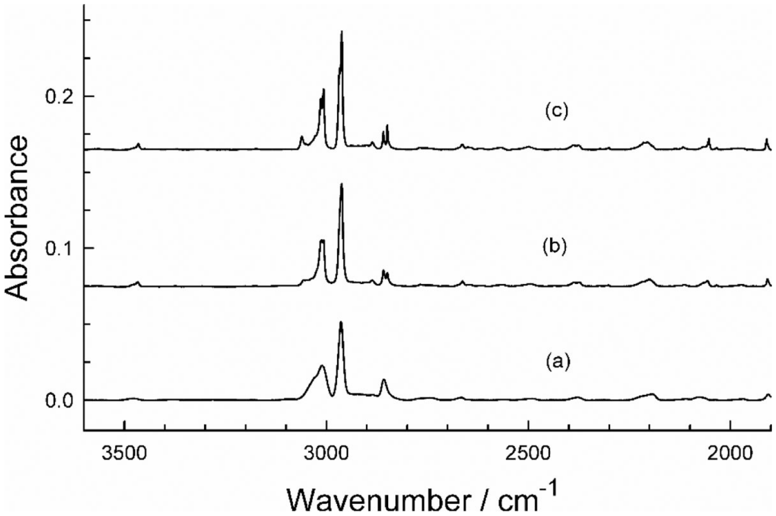

Fig. 4.

Spectra of dimethyl carbonate (a) deposited at 15 K, (b) warmed to 150 K, and (c) recooled to 15 K. Spectra have been offset for clarity.

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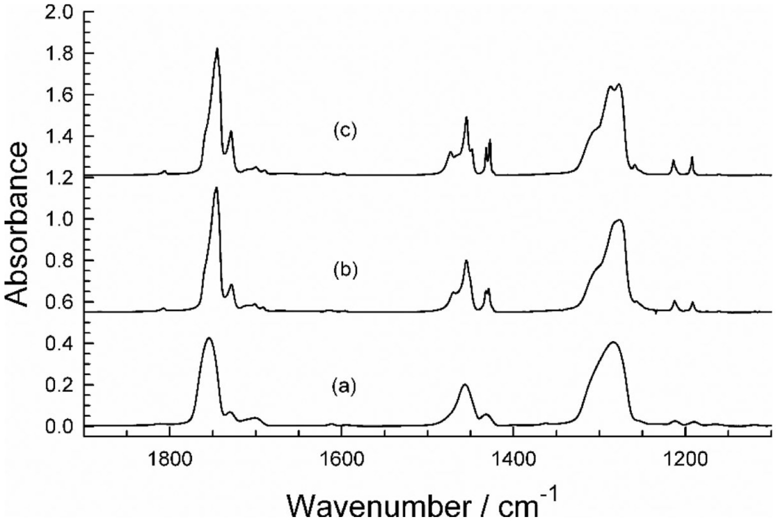

Fig. 5.

Spectra of dimethyl carbonate (a) deposited at 15 K, (b) warmed to 150 K, and (c) recooled to 15 K. Spectra have been offset for clarity.

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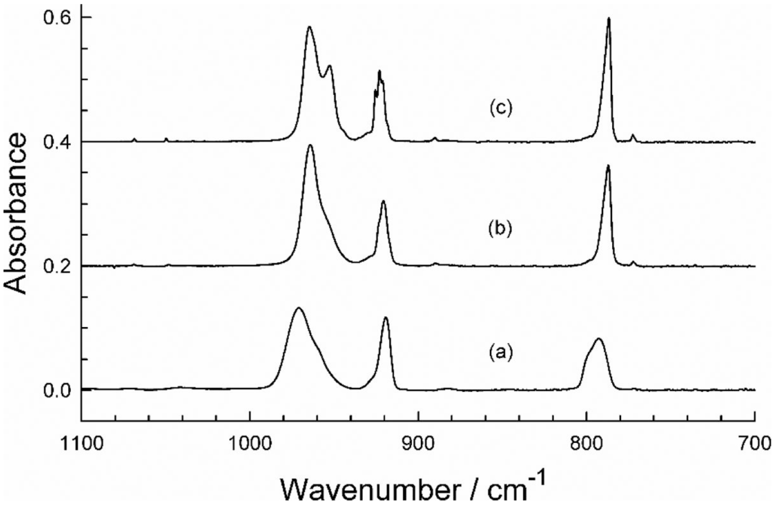

Fig. 6.

Spectra of dimethyl carbonate (a) deposited at 15 K, (b) warmed to 150 K, and (c) recooled to 15 K. Spectra have been offset for clarity.

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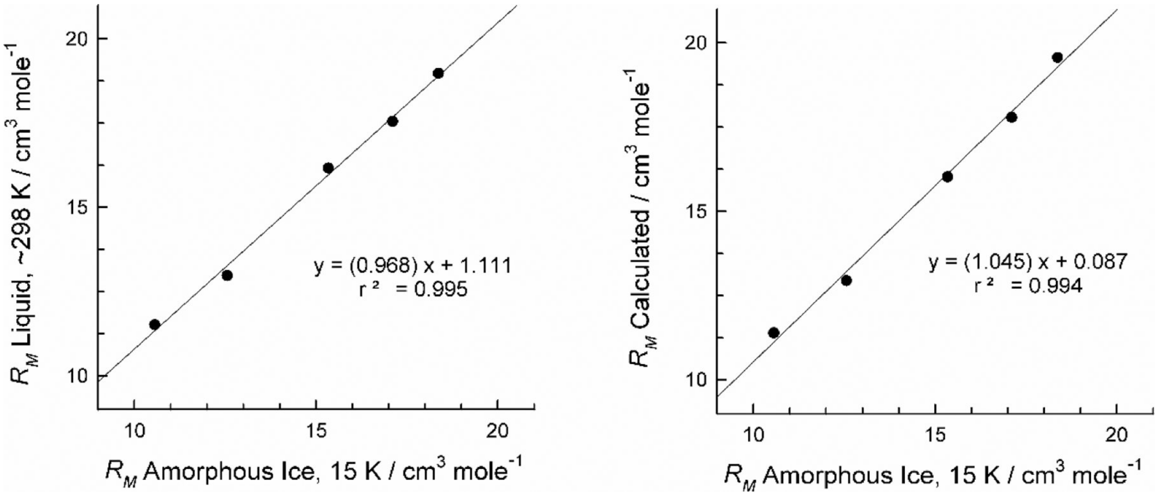

Fig. 7.

Comparisons of R_M measured for five amorphous ices at 15 K with (left) values for the corresponding liquids and (right) values calculated from bond additivity contributions. Note that the points from lower-left to upper-right in each graph follow the same order as in Fig. 1.

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Table 1 -

Refractive Indices and Densities for Five Amorphous Ices at 15 K

Property	**Acetaldehyde**	**Acetic acid**	**Acetone** [a]	**Methyl acetate**	**Dimethyl carbonate**
n	1.303	1.300	1.335	1.309	1.295
ρ / g cm^{-3}	0.787	0.892	0.783	0.832	0.903
M / g mol^{-1}	44.05	60.05	58.08	74.08	90.08
R_M / cm^3 mol^{-1}	10.56	12.56	15.34	17.11	18.37

[a] See reference 7 for more details.

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Table 2 -

Refractive Indices and Densities for Five Liquids at ~298 K[a]

Property	Acetaldehyde	Acetic acid	Acetone	Methyl acetate	Dimethyl carbonate
n	1.3316	1.3716	1.3588	1.3595	1.3687
ρ / g cm^{-3}	0.7834	1.0492	0.7899	0.9330	1.0694
M / g mol^{-1}	44.05	60.05	58.08	74.08	90.08
R_M / cm^3 mol^{-1}	11.51	12.97	16.16	17.55	18.97

[a] Taken from reference 10.

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Table 3 -

Infrared Spectral Intensities of Amorphous Dimethyl Carbonate at 15 K

Peak Position / cm^{-1}	**α' / cm^{-1}**	**Integration Limits / cm^{-1}**	**A' / 10^{-18} cm molec^{-1}**	**Approximate Description [a]**
3010	665	3066 – 2985	3.92	CH asymm stretch
2963	1439	2985 – 2940	3.64	CH symm stretch
2857	417	2873 – 2817	1.11	CH_3 def. combination
1754	13624	1785 – 1681	61.65	C=O stretch
1456	6351	1502 – 1414	26.92	CH_3 deformations
1285	12904	1340 – 1233	86.57	CO_2 asymm stretch
971	4239	995 – 937	14.04	O-CH_3 asymm stretch
919	3583	937 – 905	4.97	C-OCH_3 symm stretch
793	2556	815 – 706	5.32	CO_3 out-of-plan def.

[a] Assignments from reference 13.

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Table 4 -

Calculated and Measured Densities for Five Ices [a]

Property	Acetaldehyde	Acetic acid	Acetone	Methyl acetate	Dimethyl carbonate
Liquid R_M	11.51	12.97	16.16	17.55	18.97
Ice n	1.303	1.300	1.335	1.309	1.295
Calculated ice ρ	0.722	0.866	0.743	0.811	0.874
Measured ice ρ	0.787	0.892	0.783	0.832	0.903
% error	8.2	2.9	5.1	2.5	3.2

[a] The units for R_M are cm^3 mol^{-1} and the units for density are g cm^{-3}.

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Table 5 -

Bond Contributions to Molar Refraction [a]

Bond Type	Contribution / $cm^3\ mol^{-1}$	Bond Type	Contribution / $cm^3\ mol^{-1}$
C-H	1.69	C-O	1.51
C-C	1.25	C=O	3.38
O-H	1.73		

[a] Values taken from reference 24.

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Table 6 -

Infrared Intensities of Carbonyl Stretches in Amorphous Ices

Compound	A' / 10^{-18} cm molecule^{-1}	Source
dimethyl carbonate	61.6	This work
methyl propionate	44.8	Hudson and Mullikin, 2019 [a]
acetone	26.7	Hudson et al., 2018 [b]
methyl formate	49.6	Modica and Palumbo, 2010 [c]

[a] See reference 9.

[b] See reference 7.

[c] See reference 22.