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Infrared studies at the ice laboratory of Alcoy

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Abstract

At present, there are few laboratory spectra of analogs of astrophysical interest in the far-infrared range (FIR). Laboratory infrared (IR) spectra of simple ices and its mixtures obtained at low temperature and pressure are found mainly up to 25 μm, and few up to 200 μm. On the other hand, there are some spectra for carbonaceous material and silicates up to 2000 μm. Our laboratory is equipped with an IR spectrometer that integrates a Michelson interferometer with a resolution better than 0.25 cm^{-1} and that operates under vacuum conditions of 10^{-1} mbar. There is also a silicon bolometer, a very high-sensitivity detector in comparison with the standard deuterated triglycine sulfate (DTGS) detectors. The use of the bolometer and the possibility of working under vacuum conditions inside the optics and the sample compartment of the spectrometer allow obtaining high-sensitivity spectra free from H_2O vapor and CO_2 gas bands. Those conditions are necessary to obtain high-quality spectra in the FIR where absorption bands are much less intense than those in the mid-IR region. In our laboratory there is also a high-vacuum chamber that allows different studies on ices deposited onto a cold finger. We have already carried out experiments on the study of ice density as a function of temperature, UV irradiation of ices, temperature-programmed desorption (TPD) and UV–vis reflectance. In this work, we present the design of the experimental setup we are building to carry out different experiments simultaneously on the same ice sample, including spectra measurements in the mid-IR range (MIR) and the FIR. This design integrates jointly the IR spectrometer, the high-vacuum chamber and the silicon bolometer. Lastly, we show a spectrum we have obtained of a solid of astrophysical interest such as crystalline forsterite grains by using the polyethylene pellet technique.

Keywords: Infrared spectroscopy; Bolometer; Ices; Laboratory; Far-infrared

1. Introduction

Many laboratory spectra of ices of astrophysical interest (e.g. H_2O, CO, CO_2, CH_3OH, CH_4, NH_3) and its mixtures obtained at low temperature (10–180 K) and at low pressure ($P < 10^{-7}$ mbar) are available up to 25 μm, but only few spectra are available up to 200 μm. Laboratory spectra of cosmic analog grains (carbonaceous material and silicates) in the range 20–2000 μm have also been obtained. Therefore, there is a lack of reference spectra of ices of astrophysical interest obtained at the laboratory in the range 25–1000 μm in the far-infrared (IR) and submillimeter region.

Ice spectra and its mixtures in the FIR can be found in the literature. Far-IR spectra in the 20–200 μm range of H_2O, CH_3OH and the mixture $H_2O + CH_3OH + CO + NH_3$ was obtained by Hudgins et al. (1993). Moore and Hudson (1992) carried out spectral studies of phase changes in water ice induced by proton irradiation in the far-IR (20–100 μm). The work by Smith et al. (1994) shows the far-IR spectra of water ice in the 20–110 μm range for both the amorphous and the crystalline phase at temperatures between 10 and 150 K. Far-IR spectra of pure water ice and mixtures with simple ices such as CH_3OH, NH_3, CO, CO_2, etc. were obtained by Moore and Hudson (1994). Ion and UV irradiation of simple ices and its mixtures was carried out by Moore et al. (2001). Mennella et al. (1998) calculated the absorption coefficient of cosmic analog grains (silicate and carbonaceous grains such as fayalite, forsterite and disordered carbon grains) in the range

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20–2000 μm. On the other hand, no IR spectra of simple ices obtained at low temperature and low pressure have been published, as far as we know, in the range of 200–1000 μm.

HERSCHEL mission will provide unrivaled measurements in the relatively poorly explored 57–670 μm part of the far-IR and submillimeter spectral range bridging the wavelength gap between earlier IR missions like IRAS and ISO and the currently SPITZER and AKARI, in which all are limited to wavelengths up to 200 μm. Bands in the mid-IR range (MIR) are due to intramolecular bond vibrations being more intense than the near-IR overtones and consequently giving information of the molecular abundance. In contrast, bands in the FIR contain absorptions due to intermolecular vibrations, which give information about the transitions between phases or the lattice structure, these being bands of lower intensity than their counterparts in the MIR. Therefore, spectroscopy of high sensitivity in the FIR and submillimeter region, both in space and in laboratory, is of great interest for the astrophysical community, allowing the comparison between space and laboratory data.

One of the aims of our laboratory in the forthcoming years will be to obtain high-sensitivity spectra of molecular ices and its mixtures in MIR and FIR up to 1000 μm. We have also planned to carry out several studies simultaneously on the same sample by using techniques such as IR spectroscopy, temperature-programmed desorption (TPD), UV–vis reflectance, double-laser interference, etc. with an experimental design that it is shown in Section 2.

2. Experimental procedure

We have obtained a Fast Fourier Bruker IFS 66v/S spectrometer. It integrates a Michelson interferometer that permits obtaining spectra with a resolution better than 0.25 cm^{-1}. The basic components of this spectrometer are the source, the beamsplitter and the detector. These components must be selected depending on the spectral range under study; in our particular case, the MIR and the FIR. We have purchased the Globar$^{\mathrm{TM}}$ and the Hg-Arc sources, for the MIR and FIR, respectively; a KBr beamsplitter for the MIR and four Mylar beamsplitters (6, 25, 50 and 100 μm) covering all the FIR range. As far as the detectors are concerned, we use two deuterated triglycine sulfate (DTGS) for the MIR and the FIR, respectively, and a Si bolometer, a very high-sensitivity detector that covers the spectral range 670–10 cm^{-1} (15–1000 μm).

The major drawback with the DTGS detectors is their sensitivity: they are less sensitive than other detectors available. Another problem with these detectors is that they are slow, which means that the moving mirror inside the interferometer cannot translate very fast while an interferogram is being measured. The Si bolometer presents an increase of the detectivity of four orders of magnitude compared to the standard DTGS detectors. As a consequence, the Si bolometer lets us obtain high-sensitivity spectra using less time than the DTGS detectors. However, a difficulty with this detector is that it must be cooled with liquid helium and its dewar pre-cooled and refrigerated with liquid nitrogen, which means its use is quite complex. Moreover, liquid helium is very expensive and evaporates quickly, which implies that the experiments must be well planned in advance.

Our spectrometer operates under vacuum conditions of 10^{-1} mbar in the optics and the sample compartment. This environment makes it possible to record spectra free from gas phase interference such as H_2O or CO_2 in the MIR, and especially for measurements in the FIR where water vapor contamination is very pronounced. Fig. 1 shows the background spectra of the FIR source obtained with the DTGS FIR detector and the Mylar 6 beamsplitter under vacuum conditions in optics and sample compartment (solid line) and without vacuum conditions (dashed line). Note the strong absorptions due to water vapor in the FIR (400–30 cm^{-1}) when not working under vacuum conditions.

On the other hand, at present in our laboratory there is a high-vacuum chamber in which ices are deposited onto a substrate placed in thermal contact with a cold finger. Different experiments such as measurements of ice density versus temperature, UV irradiation of ices, TPD and UV–vis reflectance are being carried out. Fig. 2 shows an ice density measurement experiment by using the double-laser interference. At the same time a TPD experiment with a mass spectrometer connected to the bottom side of the high-vacuum chamber and a UV–vis reflectance experiment with a UV–vis spectrometer located in front of a window of the high-vacuum chamber are being carried out.

Fig. 3 shows the experimental configuration we have designed in order to obtain IR spectra of simple ices and its mixtures in the MIR and FIR. This configuration integrates the IR spectrometer, the high-vacuum chamber and the Si bolometer together. In order to offer the possibility of carrying out several experiments on the same sample at the same time, the high-vacuum chamber is located outside the sample compartment of the IR spectrometer, making available the four chamber's windows. An essential part of this design is a mechanism that permits to move the high-vacuum chamber in the three spatial dimensions by using a micrometric adjustment, which allows the right alignment of the IR beam when it travels from the IR source to the detector.

Fig. 4 shows a bottom view of our design, the IR spectrometer on the left and the high-vacuum chamber on the right. The technique used to obtain measurements is the transmission–reflection–transmission (TRT). The beam is directed to the cold finger in the chamber by using the reflection module A515. There is a vacuum of 10^{-1} and 10^{-7} mbar inside the interferometer sample compartment and the ice chamber, respectively. In order to solve the problem with the vacuum, when the beam is traveling outside and between the interferometer and the ice chamber, we use nitrogen gas.