Photodesorption of Water Ice from Dust Grains and Thermal Desorption of Cometary Ices Studied by the INSIDE Experiment

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Abstract

A new experimental setup, INterStellar Ice-Dust Experiment (INSIDE), was designed for studying cosmic grain analogs represented by ice-coated carbon- and silicate-based dust grains. With the new instrument, we can simulate the physical and chemical conditions prevailing in interstellar and circumstellar environments. The setup combines ultrahigh vacuum and low-temperature conditions with infrared spectroscopy and mass spectrometry. Using INSIDE, we plan to investigate physical and chemical processes, such as adsorption, desorption, molecule formation, on the surface of dust/ice samples. First experiments on the photodesorption of water ice molecules from the surface of silicate and carbon grains by UV photons revealed a strong influence of the surface properties on the desorption yield, in particular in the monolayer regime. In the second experiment, the thermal desorption of cometary ice analogs composed of six molecular components was studied for the first time. Codesorption of CO₂ and CH₃OH with O₂ indicates that at high O₂ concentrations in cometary or interstellar ices, “heavy” ice molecules can be partly trapped in O₂ and enter the gas phase much earlier than expected. This effect could explain astronomical detections of complex organic molecules in cold dense interstellar clouds.

Key words: astrochemistry – dust, extinction – ISM: molecules – methods: laboratory: solid state

1. Introduction

The interstellar medium (ISM) attracts our great attention as the place where stars and planets are born and from where, possibly, the precursors of life have come to Earth. The ISM is filled with a tenuous amount of materials, such as gas and dust. The dust, about 1% by total mass, consists of carbon- and silicate-based compounds (Dorschner & Henning 1995; Henning & Salama 1998; Draine 2003, 2009; Henning 2010). In the cold and dense environments of the ISM, such as dense molecular clouds and the outer parts of circumstellar envelopes and disks, dust grains are mixed with and covered by molecular ices consisting mainly of a number of simple molecules: H₂O (the major compound), CO, CO₂, NH₃, CH₃OH, CH₄, O₂, and some others, including a small amount of complex molecules, and polycyclic aromatic hydrocarbons (for reviews see Allamandola et al. 1999; van Dishoeck 2014). It is generally accepted that large bodies in circumstellar disks, such as planets, planetesimals, asteroids, and comets, are formed by aggregation of dust grains. The presence of an ice layer, particularly of polar ices, enhances the efficiency of the grain aggregation (Boogert et al. 2015; Min et al. 2016). The cometary ice composition is thought to be similar to that of the interstellar ice mantles (Mumma & Charnley 2011).

Chemical processes in the ISM can be divided into two groups, gas-phase reactions and reactions on the surface of interstellar grains. These two reaction routes interact through adsorption/desorption processes. Physical and chemical processes in/on grains are triggered by UV irradiation, cosmic rays, thermal processing, atom addition, and reactions with radicals, and lead to the formation of new molecules, and possibly, their release to the gas phase. The schematic Figure 1 shows cosmic grains mixed with molecular ices and the main sources of their processing in astrophysical environments. The influence of the structure, temperature, and composition of grains on the physical and chemical processes on their surfaces is of great importance for the general understanding of astrochemical networks, the creation of planetary systems, and the appearance of life on Earth.

Outside the sublimation distances, the dominant destructive process for the ice coverage of grains in circumstellar disks is supposed to be UV photodesorption. It was shown that photodesorption efficiently destroys water ice in optically thin disks (Grigorieva et al. 2007). Photodesorption yields have been measured for a number of ices, such as H₂O (Westley et al. 1995; Öberg et al. 2009a; Cruz-Díaz et al. 2018), CO (Öberg et al. 2007, 2009b; Muñoz Caro et al. 2010; Chen et al. 2014), CO₂ (Öberg et al. 2009b; Yuan & Yates 2013), N₂ (Öberg et al. 2009b; Fayolle et al. 2013), O₂ (Fayolle et al. 2013; Zhen & Linnartz 2014), and CH₃OH (Bertin et al. 2016). In these studies, ices were deposited on standard laboratory substrates, such as gold or KBr, that are not comparable to the dust surfaces present in the ISM.

To the best of our knowledge, the only study on the photodesorption of a monolayer of water ice coating a surface relevant to the ISM, an amorphous carbon foil, by 193 nm photons was presented by Mitchell et al. (2013). From the results presented there, one can conclude that water ice photodesorbs more efficiently from a carbon surface than from a standard substrate. What can also be mentioned in this context are the studies of the UV-induced formation of CO and CO₂ molecules on carbon grains (Mennella et al. 2006; Fulvio et al. 2012; Shi et al. 2015) and the study of the effect of an amorphous water-rich magnesium silicate on the chemical evolution of UV-irradiated methanol ices (Ciaravella et al. 2018).

There is a huge number of laboratory studies of temperature-programmed desorption (TPD) of interstellar ice analogs (for reviews see Burke & Brown 2010; Theule et al. 2013), involving desorption from silicate and carbon surfaces...
and the main sources of their processing in astrophysical environments. Ices, were presented
formation
compounds
coma of 67P vary spatially and temporally for different
Figure 1.
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spectrometry
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Figure 2. Schematic of the INSIDE experimental setup. Top right inset: UHV chamber, internal view. Bottom left inset: gas inlet system.
water ice from the surface of silicate and carbon grains and on the temperature-programmed desorption of cometary ice analogs composed of six molecular components including H2O, CO, CO2, CH3OH, NH3, and O2 from a KBr substrate, are presented in Sections 4 and 5, correspondingly.

2. Technical Description of INSIDE

The setup shown in Figures 2 and 3 has a vertical configuration and consists of two chambers separated by a gate valve. The upper ultrahigh vacuum (UHV) chamber was designed for measurements of interstellar dust/ice analogs at ultrahigh vacuum (4 × 10⁻¹¹ mbar at room temperature) and low-temperature (down to 15 K) conditions. The lower high vacuum chamber (pressure ≈10⁻⁸ mbar) is used for the annealing and extraction of dust samples without breaking the UHV conditions in the main chamber.

The low pressure in the UHV chamber is attained by pumping the chamber with a combination of a turbomolecular pump with a pumping capacity of 300 l/s (Oerlikon) backed up by a scroll pump and a NEXTorr pump (SAES) with a pumping capacity of 5001/s for H2 combining a non-evaporable-getter element with an ion pump element. The chamber has to be baked at 100°C for two weeks. Baking at higher temperatures is not possible due to the temperature limit of the coldhead. The cooling down of the sample to 15 K is achieved by mounting the sample holder on a coldhead connected to a closed-cycle helium cryostat (Advanced Research Systems).

The homemade sample holder with a KBr substrate covered by a layer of dust grains produced in another experiment (see Section 3) is moved into the lower chamber from the bottom using a magnetically coupled rotatable translator. In order to get rid of a possible adsorbate layer, the sample is typically annealed at 200°C for two hours, using a homemade annealing setup. After that, the gate valve is opened and the sample holder is moved further up and screwed on the coldhead. The translator can be removed and after closing the gate valve, the original pressure of 10⁻¹¹ mbar is reached in the chamber after about an hour.

The temperature is measured by a temperature diode permanently fixed on the coldhead. An additional diode was fixed on the sample holder near the sample and the coldhead

(Ulbricht et al. 2006; Thrower et al. 2009; Noble et al. 2012a, 2012b; Clemens et al. 2013; Smith et al. 2014, 2016; Doronin et al. 2015; Suhasaria et al. 2015, 2017; Potapov et al. 2018a). The majority of these studies are devoted to the TPD of pure ices or binary mixtures; however, interstellar and circumstellar ices are more complex. Recently, the results of TPD experiments of cometary ice analogs composed of five molecular components including H2O, CO, CO2, CH3OH, and NH3, the main constituents of interstellar and circumstellar ices, were presented (Martin-Domenech et al. 2014). The Rosetta mission to the comet 67P/Churyumov–Gerasimenko has shown that the abundance of molecular oxygen in the coma of the comet is much higher than expected and ranges 1%-10% relative to H2O, with a mean value of 3.8%. The oxygen was shown that the abundance of molecular oxygen in the coma relative to H2O, with a mean value of 3.8%. The oxygen was
Figure 1. Schematic figure showing cosmic grains mixed with molecular ices and the main sources of their processing in astrophysical environments.
diode was calibrated with respect to it. The second diode was removed after the calibration to allow the translation of the sample holder during the sample change procedure. All temperatures mentioned in the paper are the temperatures on the sample holder.

When the holder with the grain sample is fixed at the coldhead, it can be cooled down to 15 K. The coldhead is mounted onto the UHV chamber by a rotatable flange that allows us to turn the sample in different working positions. The flange is individually evacuated by a small serial turbo pump (HiPace 10, Pfeiffer) backed up by a membrane pump.

The gas inlet system of INSIDE is presented in the inset of Figure 2. It has two separate lines. The first one allows the mixing of up to five different gases. The second one is equipped with special gold seals in the leakage valves and is used for corrosive species, such as ammonia.

A schematic view of the UHV chamber is shown in Figure 4. In the present configuration of INSIDE, the following procedures can be performed:

1. Production of dust/ice samples.
   Gas mixtures can be deposited onto grains at different temperatures through two 0.5 mm diameter capillary tubes displayed in the inset of Figure 2. Two leakage valves are connected to the two different gas lines. The deposition through both lines can be done simultaneously.

2. UV processing.
   The dust/ice sample can be irradiated by UV photons. Two UV lamps separated from the main chamber by a MgF₂ window can be used: a homemade open hydrogen flow discharge lamp with a flux of 10¹³ photons s⁻¹ cm⁻² emitting at Lyα at 121 nm (10.2 eV) and a broadband deuterium lamp (L11798, Hamamatsu) with a flux of 10¹⁵ photons s⁻¹ cm⁻². The lamp has a broad spectrum from 400 to 118 nm with the main peaks at 160 nm (7.7 eV) and at about 122 nm (10.2 eV) (with the intensity of about 20% of the 160 nm peak). The signal above 170 nm is weak (about 10% of the total intensity).
   The main peaks correspond to the emission of molecular and atomic hydrogen. Such an emission spectrum can be a good analog of UV fields in interstellar clouds but also a broad UV spectrum is useful for modeling photophysical and photochemical processes in protostellar envelopes and planet-forming disks, where UV photons come from a central star having a continuous spectrum. UV fluxes of the lamps were measured in a separate experiment by the method based on the photoeffect (Fulvio et al. 2014). The uncertainty of the UV fluxes is about 30%.

3. Thermal annealing.
   The dust/ice sample can be heated up to 300 K with a controlled heating ramp rate.

4. Analytical characterization.
   (a) An FTIR spectrometer (Vertex 80v, Bruker) is used to perform in situ spectroscopy in transmission mode. The spectrometer is equipped with a MCT-detector working in the spectral range of 12,000–600 cm⁻¹. The FTIR line is separated from the main chamber by two ZnSe windows and pumped by a dry backing pump.
   (b) Species released from the sample surface can be analyzed in the gas phase by a quadrupole mass spectrometer equipped with a Faraday and a multiplier detector (multiplication factor of 10⁵) having a mass range up to 300 amu and a partial pressure sensitivity of 5 × 10⁻¹⁵ Torr (HXT300M, Hositrad).
   (c) An UV-VIS spectrometer can be coupled to the chamber, thus obtaining a third detection technique and extending the range for spectroscopic measurements.

3. Production of Dust Grain Analogs

Dust grain analogs are produced in a laser ablation setup, which is able to condense solid carbonaceous and siliceous grains and gases at temperatures between 300 and 8 K. The setup is detailed elsewhere (Jäger et al. 2008, 2009; Potapov et al. 2018a, 2018b). The gas-phase deposition of nanometer-sized amorphous grains is achieved by pulsed laser ablation of graphite or metal targets composed of Mg, Fe, and Si and subsequent condensation of the evaporated species in a quenching atmosphere of a few mbar He and H₂ for amorphous carbon grains or He and O₂ for amorphous silicate grains. The condensed grains are extracted adiabatically from the ablation chamber through a nozzle into a second low pressure chamber (10⁻³ mbar) to decouple the grains from the gas. A second extraction through a skimmer into a third chamber (10⁻⁶ mbar) generated a particle beam that is directed into a fourth, cryogenic, deposition chamber (10⁻⁶ mbar), where the grains are deposited onto a substrate. The thickness of the grain
deposit is controlled by a microbalance. The composition of the employed silicates was determined by HRTEM and EDX studies.

The grain structure can be described as amorphous. The individual particles are very small (a few nm) and the largest particle agglomerates are in the range of a few tens of nm. The morphology of dust particles on a substrate can be understood as a porous layer of rather fractal agglomerates. The porosity of the layer can be very high (Sabri et al. 2014). The surface of the dust is very large, but the area cannot be measured exactly. Figure 5 shows a SEM (scanning electron microscopy) image of silicate grains produced in our setup.

Note that carbon grains are composed of small, strongly bent graphene layers or fullerene fragments, which are linked by aliphatic bridges. Fullerene molecules of different sizes and shapes can also be contained (Jäger et al. 2008, 2009). Fullerene-like grains, as they are typically considered in the literature, are characterized by refractory and disordered carbonaceous structure. The bent graphene layers and aliphatic bridges can have a high number of defects and dangling bonds.

4. Photodesorption of Water Ice from the Surface of Dust Grains

Figure 6 shows the IR transmission spectra of MgFeSiO$_4$ and $^{13}$C grains deposited on KBr substrates, the samples used in the present study. The spectra were taken in the UHV chamber of INSIDE after their exposure to air and annealing in the lower chamber of INSIDE. In the silicate spectra, one can see the band related to the Si–O stretching vibration at 990 cm$^{-1}$ and additional bands due to interaction with air: around 3500 cm$^{-1}$ corresponding to O–H stretching in associated Si–OH groups and around 1500 cm$^{-1}$ corresponding to magnesium carbonate. In the carbon spectra the assignable bands are due to C–H stretching vibrations corresponding to the saturated –CH$_3$ and –CH$_2$ groups, C=O and C=C stretching, and C–H deformation.

For these experiments, 3 and 66 monolayers (ML) of water ice were deposited onto the surface of amorphous MgFeSiO$_4$ and amorphous $^{13}$C grains at 15 K (assuming the surface area corresponding to the KBr substrate area of 1 cm$^2$ and a monolayer column density of $1 \times 10^{15}$ molecules cm$^{-2}$). The deposition rate was about 10 ML minute$^{-1}$. The thickness of the water layer was determined from the 3,07 mm water band $T_{LO} = 10$ cm$^{-1}$, with the water layer determined from the IR transmission spectra before and after the UV irradiation of H$_2$O ice deposited on carbon grains at 15 K.

An empirical dependence of the photodesorption yield on the temperature (T) and ice thickness (x) $Y_{pd} (T, x) = 10^{-3} (1.3 + 0.032 \times x)^{0.3} \times (1 - e^{-x/\lambda(T)})$, where $\lambda$ is a temperature-dependent ice diffusion parameter, was determined previously (Öberg et al. 2009a). Using this dependence, we obtained the values for the
The photodesorption yield of H$_2$O at 15 K of $\sim 1.8 \times 10^{-3}$ and $1.7 \times 10^{-3}$ molecule/photon for 66 and 3 ML correspondingly. Thus, the experimental values of the photodesorption yields for 66 ML of H$_2$O on MgFeSiO$_4$ grains is in agreement with the literature data. The binding energies of H$_2$O molecules on amorphous silicates and standard substrates, such as CsI and gold, experimentally determined using the TPD technique were found to be quite similar varying within a value of about 15% (Penteado et al. 2017). Thus, our result indicates that water ice is weakly bound on the surface of silicates. The much lower yield for 3 ML can be explained by a very large surface of grains that can be one to two orders of magnitude larger than the nominal surface of the substrate, on which grains are deposited (Potapov et al. 2018a). Therefore, we probably have a sub-monolayer ice coverage of the dust instead of “nominal” 3 ML. Using the thickness of 0.3 ML instead of 3 ML in the empirical equation above, we obtain a photodesorption yield of 0.5 in agreement with our experimental value.

The photodesorption yield for H$_2$O on carbon grains is noticeably higher compared to silicate grains. For 66 ML, it is still in the limits of the total experimental uncertainty. However, for the 3 ML it is nine times higher compared to MgFeSiO$_4$ grains and three times higher compared to the literature data. A reason for this finding could be the differences in the surface properties of carbon and silicate grains. The fullerene-like carbon grains used in the present study are typically considered to be hydrophobic, whereas silicates are known to have hydrophilic and hydrophobic surface groups. The finding of stronger bound water molecules to silicate grains in comparison to fullerene-like carbon grains was demonstrated in our recent study (Potapov et al. 2018a). However, this result needs further investigations.

In the experiments with carbon grains, the formation of $^{13}$CO$_2$ was observed (see Figure 9). This result confirms that the dust surface is active. The formation of CO and CO$_2$ by UV irradiation of water ice covering the surface of carbon grains or layers is already well-known (Mennella et al. 2006; Fulvio et al. 2012; Shi et al. 2015). It was also demonstrated that other energetic processes, such as ion and proton bombardment, lead to the same result (Mennella et al. 2004; Raut et al. 2012; Sabri et al. 2014). The addition of O/H atoms onto the surface of bare carbon grains makes one more step toward the molecular complexity leading to the formation of H$_2$CO (Potapov et al. 2017). The formation cross section of CO$_2$ obtained in our study following the procedure described in Mennella et al. (2006) is $0.3 \pm 0.04 \times 10^{-18}$ cm$^2$ and is in good agreement with the values provided there.

5. TPD of Pre-cometary Ice Composed of Six Components

For these experiments, ices composed of five (H$_2$O, CO, CO$_2$, CH$_3$OH, NH$_3$) and six (H$_2$O, CO, CO$_2$, CH$_3$OH, NH$_3$, and O$_2$) molecules were deposited at 15 K onto a KBr substrate. H$_2$O, CO, CO$_2$, CH$_3$OH, and O$_2$ were premixed and NH$_3$ was deposited simultaneously with the mixtures through the separate line of the gas inlet system. We tried to reproduce the mixing ratios (by number of molecules) observed for the comet 67P/Churyumov–Gerasimenko (Altwegg et al. 2017; K. Altwegg 2019, private communication). There is remarkable diversity in the mixing ratios of molecular ices in comets. Abundances for each species with respect to H$_2$O range between one and two orders of magnitude (Dello Russo et al. 2016). We chose the comet 67P as the most recent object. The mixing ratios for 67P and used in our experiments are presented in Table 2. Figure 10 shows a spectrum of a mixture of six ices. The vibrational bands of H$_2$O, CO, CO$_2$, CH$_3$OH, and NH$_3$ are clearly observable and listed in Table 3. The ice thicknesses were determined from the IR band areas using the corresponding band at 1559 cm$^{-1}$ (Ehrenfreund et al. 1992) was not observed. Therefore, the mixing ratio of O$_2$ was
estimated only from the O₂ pressure in the gas mixture before the deposition.

After the depositions, TPD experiments were performed by linear ramping of the sample temperature with a rate of 2 K minute⁻¹ in the temperature range between 10 and 200 K. Infrared spectra were measured during the warming-up in the spectral range from 6000 to 600 cm⁻¹ with a resolution of 2 cm⁻¹. Mass spectra were taken during the warming-up in the mass range from 0 to 90 amu with a scanning time of one minute for one spectrum.

A very detailed comparison of the TPD behavior of a five-component ice mixture to the TPD behavior of its individual components was done recently (Martin-Domenech et al. 2014). Here we compared five- and six-component ice mixtures to understand if there is an influence of the additional O₂ in the mixture on the desorption behavior of the composing molecules following the discussion in the introduction.

The MS TPD curves obtained during the warming-up of the mixture of six ices containing 4% of O₂ (Exp3 in Table 2) are presented in Figure 11. Partial pressures are presented for six masses: 17 (OH and NH₃), 18 (H₂O), 28 (CO), 31 (CH₃OH), 32 (O₂ and CH₃OH), and 44 (CO₂).

Variations in the TPD peak positions for the ice mixtures containing 0%, 1%, and 4% of O₂ are in the limits of 1 K, which can be addressed to experimental uncertainties and measurement errors. However, a noticeable shift of the O₂ signal and additional TPD peaks are observed for the ice mixture containing 10% of O₂. TPD peak temperatures, corresponding molecules, and desorption types are listed in Table 4. All temperatures are in agreement with the literature data (Collings et al. 2004; Martin-Domenech et al. 2014). TPD peaks at temperatures above the desorption temperatures corresponding to the pure ices are explained by trapping of species in the water-rich matrix. The NH₃ is indistinguishable from OH, which has, in addition, a much stronger signal. Additional TPD peaks indicate that at high O₂ concentrations in cometary or interstellar ices, "heavy" ice molecules, such as CO₂ and CH₃OH, can be partly trapped in O₂ and release to the gas phase together with O₂ at temperatures much lower than expected. The amount of O₂ molecules desorbing independently relative to the amount of molecules desorbing due to their trapping in water matrix (volcano desorption and codesorption) increases from ~10% for the 1% O₂ mixture to ~60% for the 10% O₂ mixture. The amounts of CH₃OH and CO₂ molecules co-desorbing with O₂ relative to the H₂O-related desorption are about 2% and 1% correspondingly.
indicating that water molecules are weakly bound on the surface of silicates. The much lower yield for 3 ML of H₂O ice on MgFeSiO₄ grains is in agreement with our previous study, which demonstrated that ice molecules desorb from a surface of grain clusters that is much larger than the nominal surface of the substrate. The photodesorption yield for H₂O on carbon grains is noticeably higher compared to silicate grains. This result could be explained by the hydrophobic surface properties of the carbon grains.

Temperature-programmed desorption of a six-component ice (H₂O, CO, CO₂, CH₃OH, NH₃, and O₂) was measured for the first time. No changes in the TPD peaks positions of the ice components due to an addition of up to 4% of O₂ to the ice mixture have been observed. Codesorption of CO₂ and CH₃OH with O₂ in the ice containing 10% of O₂ indicates that at high O₂ concentrations in cometary or interstellar ices “heavy” ice molecules can be partly trapped in O₂ and release to the gas phase much earlier than expected. This effect could explain astronomical detections of complex organic molecules in cold dense interstellar clouds.

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**Table 4**

| TPD Peak temperature, K | TPD Peak temperature, K | Molecule | Desorption Type  |
|------------------------|------------------------|----------|-----------------|
| CH₃OH                  | CH₃OH                  | Codesorption with O₂ | CH₃OH |
| H₂O                    | H₂O                    | Codesorption with O₂ | H₂O |
| CO                     | CO                     | Volcano desorption of molecules trapped | CO |
| CO₂                    | CO₂                    | In water ice matrix due to the transformation of the water ice structure from amorphous to crystalline | CO₂ |
| CH₃OH                  | CH₃OH                  | Codesorption with H₂O | CH₃OH |

**Notes.**

a Variations in the TPD peak positions for different ice mixtures are in the limits of 1 K, which can be addressed to experimental uncertainties and measurement errors.

b For such complicated mixtures with many interactions between molecules in the water ice matrix, which can have a high porosity and consequently a large surface, it is hard to distinguish between monolayer and multilayer desorption.

c For methanol, this gives ≈2 × 10⁻³ CH₃OH molecules per O₂ molecule, which is much more than the amount of methanol co-desorbing with CO (Ligerink et al. 2018). Thus, co-desorption of molecules with O₂ could partly explain the detection of CH₃OH (Vastel et al. 2014; Potapov et al. 2016) and other complex organic molecules in cold dense molecular clouds, if we assume a relatively high amount of O₂ (similar to the comet 67P) in interstellar ices.

**6. Conclusions**

A new experimental setup has been developed in our laboratory and described in this paper. INSIDE allows the studying physical and chemical processes on the surface of bare or ice-covered cosmic dust grain analogs. Photodesorption of water ice molecules deposited onto the surface of amorphous silicate and carbon grains was measured for the first time. The photodesorption yield for 66 ML of H₂O ice on MgFeSiO₄ grains is in agreement with the literature data
