Study of the reactivity between carbon dioxide and ethylamine on interstellar dust grains

Internship Report

May 2nd – June 23rd, 2023

Figure 1 :Carina Nebula North

Image Credit & Copyright: [Carlos Taylor](#)

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Acknowledgments

First, I would like to thank LERMA Cergy's director François Dulieu, for granting me this internship opportunity. Discovering the field of astrochemistry, and even though my resume was honestly catastrophic, and my cover letter pretty bland, he still gave me a chance. Mr. Dulieu also always answered questions in a very complete way, which allowed for a better understanding of subjects, even those that I thought I had already understood. Other interns and I also had the opportunity to visit the Paris Observatory with Mr. Dulieu, which was a very interesting experience.

I would also like to thank Julie Vitorino, the PhD student at LERMA with whom I spent my time until now. From my very first day, Julie has always been encouraging, allowing me to gain confidence in my skills as well as to feel useful in the laboratory. Julie also gave me a lot of autonomy in the realisation of the experiments which was very rewarding. Working day after day with Julie was very enjoyable, as she would share her reflexions, and give me the chance to give my input.

I also have to mention Jessica Ferrero, a visiting PhD student who was very welcoming, and took some of her time to explain to me what her quantum chemistry calculations were about and showing me what it looked like.

And of course, everyone else at LERMA Cergy for making this internship a good time. The atmosphere in the laboratory is great, and every professor is very involved, always willing to explain anything I would not understand.

It was a great first month of internship, and I can only hope that the next one will be as good.

Thank you.

Introduction

The interstellar medium (ISM) is the matter and radiation that exist in space, between star systems within a galaxy. In this medium, more than 240 different molecules[1] have been observed, with some of them, such as formamide, believed to be molecules involved in the apparition of life[2]. Therefore, it is crucial for astronomers to try and understand how these molecules are formed, and how they can react together, to further improve our comprehension of chemistry, but also to try and answer the thousands year old question: *"Where does life come from?"*

Within the ISM a huge chunk of the chemistry happens in molecular clouds, which are parts of the ISM that have higher density than average[3], in which molecules (such as H_2, the most common one) can form. Despite the density being higher in molecular clouds than in the rest of the ISM, it is still very low. Therefore, a significant portion of chemical reactions happen at the surface of interstellar dust grains. Indeed, with the atom density being so low, the probability of atoms colliding with each other is very low, whereas they can more easily meet at the surface of dust grains after being adsorbed on it. Therefore, these interstellar dust grains play the role of catalysts in these reactions.

Being able to reproduce the thermodynamical conditions found in molecular clouds, and to make experiments on it is very important to better understand what leads to the apparition of complex organic molecules, as well as to confirm observations from astronomers, and gives us a better chance to one day, understand the mechanisms that lead to favourable conditions for the apparition of life.

To this effect, the researchers at LERMA Cergy, led by Pr. François Dulieu have developed experiments to recreate these conditions. During a set of experiments in September of 2022, some results showed that ethylamine and carbon dioxide had reacted together. Recreating these experiments and identifying the product as well as the conditions for the reaction will be the focus of my internship.

1. Presentation of LERMA Cergy

1.1. General presentation

LERMA is a French public research unit founded in 2002.

It spreads across the CNRS, Paris observatory, Sorbonne universités and CY Cergy Paris université.

It is divided in four branches, which are:

-Galaxies and cosmology

-Dynamics of the interstellar mediums and stellar plasma

-Molecules in the universe

-Instruments and remote sensing

LERMA CY is part of the "Molecules in the universe" branch. LERMA CY is an empirical research laboratory which studies the formation of molecules on cold surfaces -i.e, interstellar dust grains, as well as other processes such as adsorption, desorption and diffusion.

To this effect, the laboratory has developed two experimental apparatuses over the years, FORMOLISM (FORmation of MOLecules in the InterStellar Medium), as well as VENUS (VErs de NoUvelles Synthèses), and is working towards FORMOLISM 2, which should replace FORMOLISM from 2023's fall. The main goal of these apparatuses is to recreate the conditions in the ISM to study molecular behaviours in these conditions. During my internship, I worked on the VENUS apparatus, and will give a presentation of the setup further down this report, as it is essential to understand the experiments, and the work which has been done.

1.2. Current team

The team is led by my tutor, Pr. François Dulieu, joined by five associate professors with backgrounds in chemistry and physics. There is one research engineer, one computer science engineer and one technician. Finally, four Ph.D. students are part of the team, in particular Julie Vitorino with whom I worked during my internship.

2. Presentation of the VENUS apparatus

VENUS is an ultra-high vacuum[4] experimental setup designed to simulate the pressure and temperature conditions found at the surface of interstellar dust, to study adsorption, reactivity and thermal desorption at the surface of interstellar dust.

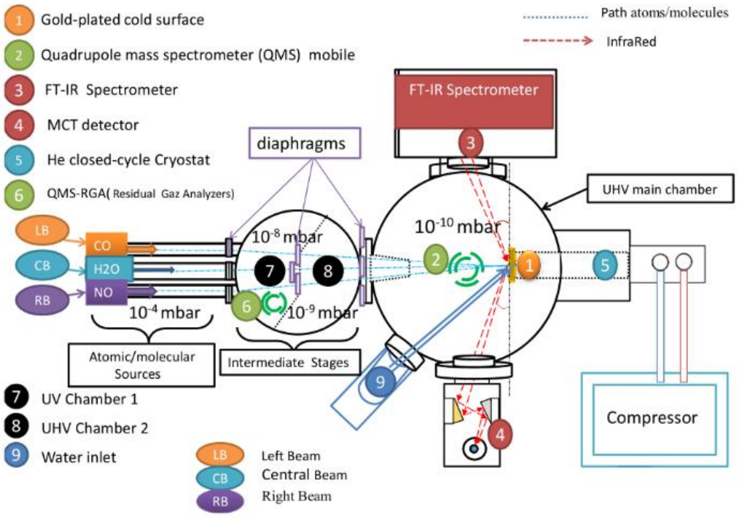


Figure 2: Schematic top view of VENUS

VENUS is constituted of three chambers: The main chamber with the lowest pressure, and two intermediate chambers, at slightly higher pressures. These chambers are separated from one another by diaphragms, and the entirety of the compound is made of stainless steel to prevent corrosion.

The main chamber is the one in which experiments are conducted. Ideally, its pressure is lower than 2.10^{-10}mbar to properly recreate the conditions of the ISM. In the middle of the chamber there is the sample holder, a circular gold-plated surface, with a radius of 9mm, which can be cooled down to 6K by a cryostat. The cryostat uses gaseous Helium turned liquid and producing the cold through adiabatic expansion. It can be heated up to around 400K through a resistive heater. It is on this surface that every study will be conducted.

The studied species are propelled on this gold substrate through the chambers by three molecular beams, allowing us to send several species at the same time, or to alternate without needing to replace products every time. These beams can be connected to liquid species, gases whose flow can be regulated by an automated flow regulator or needle valves. Water has its own special inlet directly into the main chamber. To ensure that the correct product is sent through the beams, the beams as well as the routing circuit are pumped upon before experiments, and the output of each beam is tested through mass spectroscopy.

Finally, there are the analysis instruments. For the main chamber an InfraRed (IR) spectrometer is located outside of the chamber, with the mid-IR source being located on one side of the chamber, the beam is then reflected on the sample holder and a mercury cadmium telluride detector on the opposite side of the chamber. The detector must always be cooled down with liquid nitrogen. Along with the IR spectrometer, a Quadrupole Mass Spectrometer (QMS) is located in the main chamber, in front of the sample holder. It can be moved to a low position during to not be in the pathway of the molecules during deposition times, and to a high position during Thermal Programmed Desorption (TPD) (See 3.2.) to analyse the desorbed molecules, as well as while testing the beams. It is also used before experiments to quantify the abundance of residual gases in the chamber. Another mass spectrometer is located in the first chamber and can be used to check the steadiness of beams during the deposition.

3. Surface chemistry and physics

3.1. Adsorption, diffusion and desorption

Adsorption is the adhesion of species to a surface. Two types of adsorptions exist: chemisorption and physisorption. The classification of adsorption between these two types depends primarily on the adsorption energy of the adsorbed specie to the surface. Physisorption is weaker and usually involves Van der Waals interaction, while chemisorption involves chemical bonds.

Diffusion is the ability for an atom or molecule to move from one point to another of the surface.

Desorption is the opposite process of adsorption. It happens when the molecule reaches energy higher to its adsorption energy that keeps it bound to the surface. When a molecule desorbs due to temperature, it is called a thermal desorption, and in our case the molecule goes back to the gas phase.

3.2. Reaction mechanisms

On the substrate, two major mechanisms[5] can lead to reaction between molecules:

The first one is the Eley-Rideal mechanism, in which one molecule is adsorbed to the surface, while the other one reacts with it directly coming from the gas phase, without being adsorbed to the surface.

The Langmuir-Hinshelwood mechanism on the other hand involves both molecules adsorbing to the surface, and at least one of them diffusing on the surface to meet the other one. Diffusion is only possible through thermal agitation or through quantum tunnelling, therefore this mechanism is more likely to intervene at higher temperatures, or with higher energies.

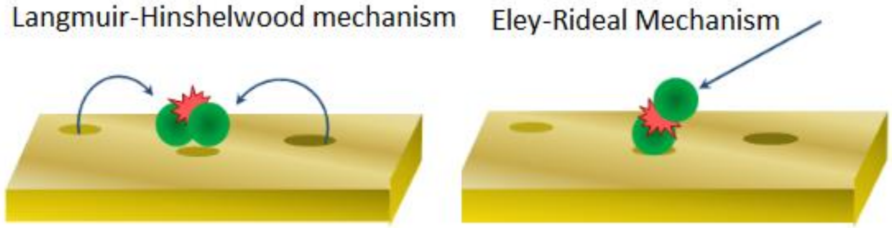


Figure 3: Schematic representation of the Langmuir-Hinshelwood and Eley-Rideal mechanisms. Illustration by Julie Vitorino

3.3. Thermal Programmed Desorption (TPD)

Thermal Programmed Desorption[6] is a method to study thermal desorption of molecules from the sample holder. During a TPD, the temperature is increased linearly with a given heating rate, usually 12K/min or 9K/min in our experiments. During the heating phase, the QMS is placed in front of the sample holder which allows us to continuously record the mass spectra of desorbing species at each temperature. The thermal desorption of an absorbed species from the surface follows an Arrhenius law described by the Polanyi-Wigner equation:

$$r(N, E_b, T) = -\frac{dN}{dT} = AN^n e^{-E_b/k_B T}$$

With:

r: the desorption rate (molecules.cm^{-2}.s^{-1})

E_b: the adsorption energy (K) which depends on our molecule

A: the pre-exponential factor (s^{-1})

N: the number of absorbed atoms or molecules

n: the order of the desorption process

k_b: the Boltzmann constant (1,38.10^{-23} J.K^{-1})

T: the desorption temperature (K)

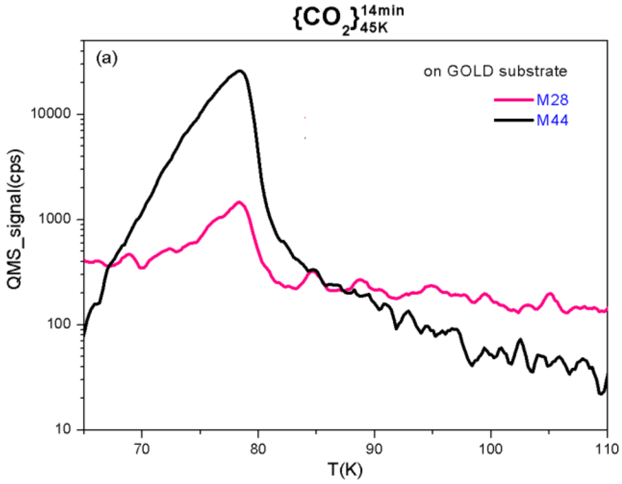


Figure 4: TPD plot corresponding to the desorption of pure CO_2 after a deposition of 14min at 45K. Logarithmic scale

When a molecule is desorbed, it hits the QMS's head. The QMS will ionize the molecule, and detect the m/z ratio, where m is the mass of the ion and z is the charge of the ion. Usually, z is equal to 1 and therefore we will refer to a detected m/z ratio simply as a mass. During this process, the molecule can sometimes be fragmented, and therefore be detected through several masses (M). For example, carbon dioxide can be associated with M44 corresponding to $CO_2^{+\bullet}$ and M28 corresponding to $CO^{+\bullet}$. The different masses associated with a molecule, coupled with their detection intensity, form together the cracking pattern of a molecule. For example, CO_2's cracking pattern is 88% M44, 4% M28 and 8% other masses. When several molecules can be associated with the same mass, the cracking patterns will help us associate the signal to the correct molecule.

4. Reactivity between ethylamine and carbon dioxide

4.1. State of the art

During my internship, the reactivity between ethylamine $C_2H_5NH_2$ and carbon dioxide CO_2 is studied. As the experiments are still ongoing, it is hard to give a deep interpretation of the results. Therefore, this part will focus on describing the experiments, as well as explaining a few results.

The reactivity between amines and carbon dioxide is well studied. CO_2, as a greenhouse gas, is one of the main causes of global warming, and a lot of CO_2 capture processes[7,8], to cancel its greenhouse gas properties, involve amines, such as aminomethanol NH_2CH_2OH.

In the case of astrochemistry, the possible synthesis of amino acids, such as glycine or alanine through CO_2 reactivity with amines is researched. Researchers from the Aix-Marseille University have already studied the reactivity between ammonia NH_3 and carbon dioxide[9] and showed the formation of ammonium carbamate $[NH_2COO^-][NH_4^+]$, as well as carbamic acid dimer $(NH_2COOH)_2$. They also overserved the formation of methylammonium methylcarbamate $[CH_3NHCOO^-][CH_3NH_3^+]$ from methylamine CH_3NH_2 and carbon dioxide[10] while trying to create glycine.

There are 2 goals to our set of experiments. First, it is to see if we can manage to form glycine, or alanine in our conditions. Second, is to understand reactions observed from the researchers at Aix-Marseille university can be observed with ethylamine and carbon dioxide, what product(s) would be formed and what are the optimal conditions for its/their formation.

4.2. Some experiments and analysis

Each experiment follows the same two steps: Each of the reagents is deposited one after the other on the substrate for a given time, at a given temperature. Several reagents can also be deposited at the same time, this is called a codeposition. The depositions can be monitored by IR spectroscopy, allowing to see the growth of the reagent's peaks, as well as noticing the apparition of other bands, related to products. The integration of the area of the peaks from one of the reagents allows to compare to a reference deposition, and therefore to estimate the amount of reagent consumed by the reaction, if there is one already at deposition temperature.

After the deposition is over, the QMS is placed in front of the cold surface, and a TPD starts. This allows us to detect preselected masses, and therefore study the desorption of products in function to temperature.

During different experiments will vary several parameters, such as the temperature of deposition, the flux of reagents relative to one another, whether the reagents are deposited at the same time or one after another, etc. These different conditions are used to narrow the conditions that are optimal for the reaction(s) to occur.

Carbon dioxide will be identified through its most intense mass M44 as well as secondary mass M28, while for ethylamine the most intense mass will be M30, and M44 and M45 will be secondary masses.

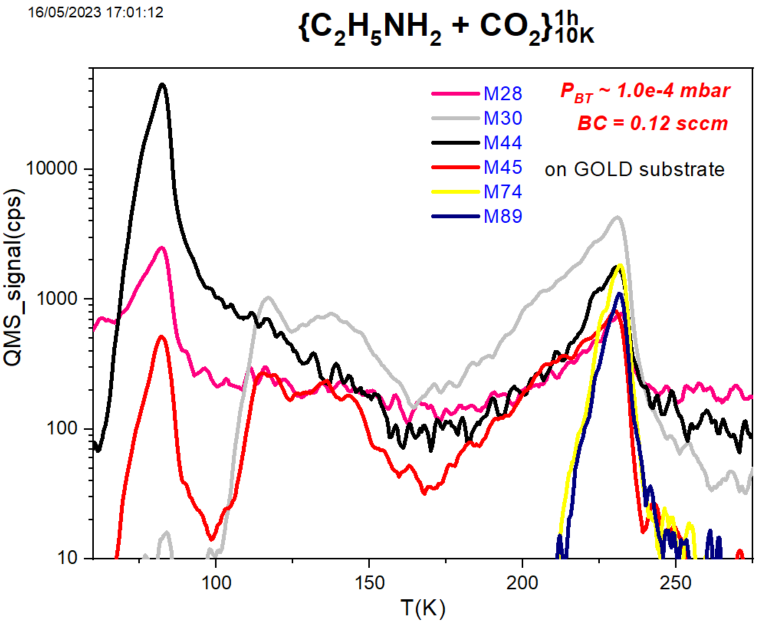


Figure 5: TPD plot corresponding to the desorbing molecules after a codeposition of ethylamine and carbon dioxide during 1 hour at 10K. Logarithmic scale

Figure 5 corresponds to one of the earliest experiments ran. Both ethylamine and carbon dioxide were deposited on the surface at the same time, with a similar flux, at a surface temperature of 10K.

From this plot, we can see that an amount of CO_2 desorbs at its normal desorption temperature of 70-100K through its main mass M44, as well as an amount of ethylamine at its normal desorption temperature of 110-150K through its main mass M30. Then by continuing the TPD, we observed that around 210-240K, more molecules desorbed, notably through M89, which is the sum of both parent masses of CO_2 and ethylamine, and M74, which is probably the same molecule with the loss of a $\bullet CH_3$. With these masses, we can also see the beginning of a second desorption of M30. This latter desorption of M30 happens simultaneously with the desorptions of M44 and M45, and with approximately the same values relative to each other than during the desorption of ethylamine at 110-150K. This leads us to think that some amount of ethylamine is also desorbing at 210-240K.

These results seem could lead to think thar our product is a ethylammonium ethylcarbamate salt $[C_2H_5NHCOO^-][C_2H_5NH_3^+]$, similar to those mentioned in the beginning of this section.

Due to its presence on interstellar dust grains, and as it was enhancing the reaction of formation of methylammonium methylcarbamate in the experiments of J-B. Bossa from Aix-Marseille University, it was important for us to replicate our experiments with water.

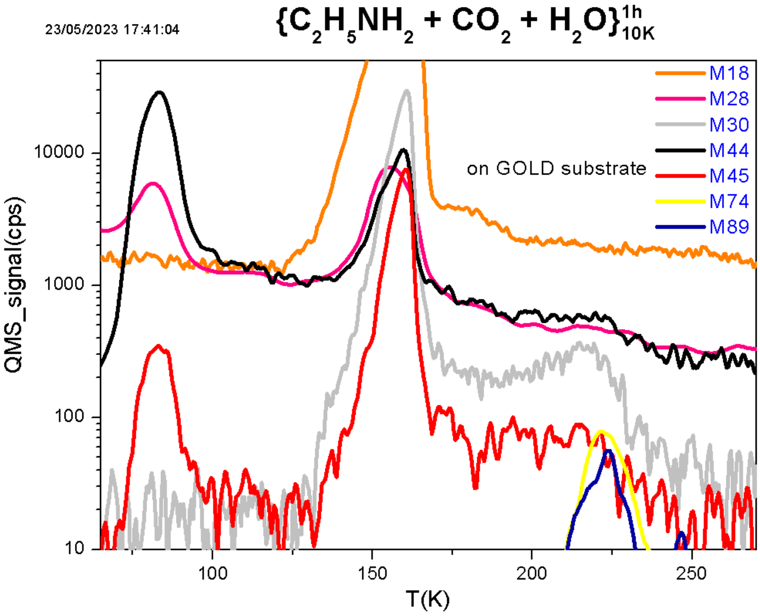


Figure 6: TPD plot corresponding to the desorbing molecules after a codeposition of ethylamine, carbon dioxide and water during 1 hour at 10K. Logarithmic scale

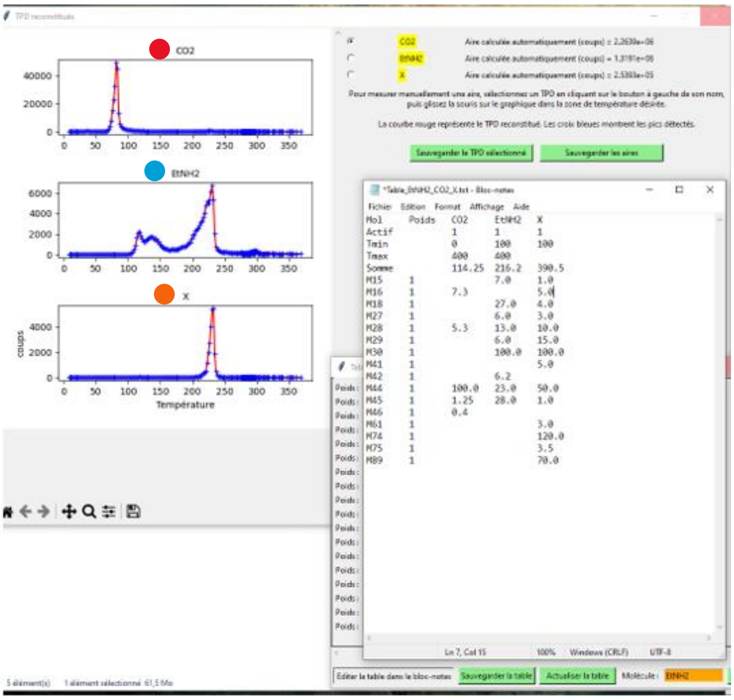
Figure 6 is the TDP from an experiment with the same conditions as the previous one for the reagents, except that we also deposited water at the same time. The water flux is around two times and a half higher than those of CO_2 and ethylamine.

In presence of water, there is almost no reactivity. In effect, the amount of product formed (M74 and M89), is around ten times lower. This lower product amount goes hand in hand with a smaller ethylamine (M30) desorption at 210-240K, also by a factor of around ten, which could reinforce the idea that the ethylamine was forming a salt with our carbamate and desorbing at the same time. Smaller desorption of ethylamine at 210-240K also leads to a more important desorption of ethylamine earlier. The other things to see are that the pure ethylamine peak shifted towards a higher temperature and desorbs together with water (M18) at 130-160K. This is because the ethylamine interacts strongly with water and will only detach from water when water itself desorbs 130-160K.

All these observations have been repeated with the same experiment performed at 55K. The effects of water on our reaction seem very different than those seen for methylamine with carbon dioxide from Aix-Marseille University. This could be explained by the affinity of ethylamine with water which would prevent the reaction, but more experiments need to be done, such as depositing a layer of water, then a layer of CO_2, then a layer of ethylamine, to see if isolating the ethylamine from water would change its behaviour.

To confirm whether our product is ethylamonnium ethylcarbamate, I also used a software developed in-house that, from experimental TPDs, tries to fit theoretical TPDs with given cracking patterns. After experimentally determining the cracking pattern of pure carbon dioxide and pure ethylamine, I could try, by trial and error, to determine the fragments involved in the product to deconvolute the signal to fit our experimental data.

After properly calibrating, this software allows us to reconstruct a molecule's desorption given its fragmentation by our QMS, and to verify the molecules responsible for the signal that we detect. From the curves on Figure 7, we can see that according to the software, no CO_2 (●)is desorbing at high temperature, our product (●) is only desorbing at high temperature, and ethylamine (●) is desorbing both at its usual temperature, as well as part of it desorbing with the product. These results could be explained by the creation of an ethylammonium ethylcarbamate compound which would cause the ethylammonium part to desorb at the same time as the ethylcarbamate.



◉ CO2 Aire calculée automatiquement (coups) = 2.2630e+06
○ EtNH2 Aire calculée automatiquement (coups) = 1.3191e+06
○ X Aire calculée automatiquement (coups) = 2.5303e+05

Pour mesurer manuellement une aire, sélectionnez un TPD en cliquant sur le bouton à gauche de son nom, puis glissez la souris sur le graphique dans la zone de température désirée.

La courbe rouge représente le TPD reconstitué. Les croix bleues montrent les pics détectés.

```
Mol     Poids   CO2     EtNH2   X
Actif           1       1       1
Tmin            0       100     100
Tmax            400     400
Somme           114.25  216.2   390.5
M15     1               7.0     1.0
M16     1       7.3             5.0
M18     1               27.0    4.0
M27     1               6.0     3.0
M28     1       5.3     13.0    10.0
M29     1               6.0     15.0
M30     1               100.0   100.0
M41     1                       5.0
M42     1               6.2
M44     1       100.0   23.0    50.0
M45     1       1.25    28.0    1.0
M46     1       0.4
M61     1                       3.0
M74     1                       120.0
M75     1                       3.5
M89     1                       70.0
```

Figure 7: Screenshot from the TPD simulator, applied to {CO_2 + $C_2H_5NH_2$} 10K 1hour. Right side: experimental cracking patterns. Left side: Reconstructed TPDs

4.3. Conclusion of the experiments

For now, the main takeaways from the experiments are that we do have reactivity between carbon dioxide and ethylamine. The product formed is not alanine but is likely to be an ethylammonium ethylcarbamate $[C_2H_5NH_4^+][C_2H_5NHCOO^-]$ compound. Further analyses, notably of the fragments and IR spectra, would confirm it. The optimal conditions for the reaction need to be worked on, as at first glance, the quantity of product depending on the temperature of deposition is not standard. The presence of water also seems to prevent the.

Conclusion

Although my internship is not over, I can confidently say that it will end up being the most interesting part of my bachelor's degree and the one during which I grew the most.

The most important thing it made me realise that chemistry, and more generally science is built by experiments and observations, and not the other way around. During my bachelor's degree my focus has always been on theory, and I didn't appreciate lab practice much. During my stay at LERMA, I discovered how much theory and experimentation go hand in hand, that you can build a theory based on results from experiments, as well as verify theories through experiments.

I also had the opportunity to have a lot of autonomy in the experiments, which I am very grateful for and gave me a bit of confidence, as before the beginning of the internship I could not fathom how I, a bachelor's student, could really be useful in a research laboratory. While I honestly can't say I was of much help on a theoretical point of view, I think my motivation to learn how the experiments worked in practice allowed me to get started quickly, and in the end I was able to perform experiments from the preparation to the data processing part, which is very rewarding in my eyes. I feel like these practical skills, while they may not translate as much to another field, have also allowed me to understand how researchers are able to get reliable results, by the control of the parameters through a lot of preparation every day.

Finally, this internship allowed me to see a decent part of what being a PhD student is like, with what day to day work looks like, the rigor required in publishing an article, or what a doctoral mission could be. In Julie Vitorino's case it is a mission of transmission of knowledge, and I had the opportunity to be part of the astronomy day organised, which allowed students ranging from primary school to high school to visit laboratories at the university as well as participating in several workshops on the theme of astronomy. As someone who thinks that the implication of the teacher is the most important thing in teaching, it was a very interesting day to be a part of.

To conclude, I want to reiterate how grateful I am to have had the opportunity of this internship, as I learned a lot about experimental astrochemistry, but also as I feel it made me grew as a scientist. I was very lucky to find such an encouraging environment, in a domain that I greatly enjoy.

References

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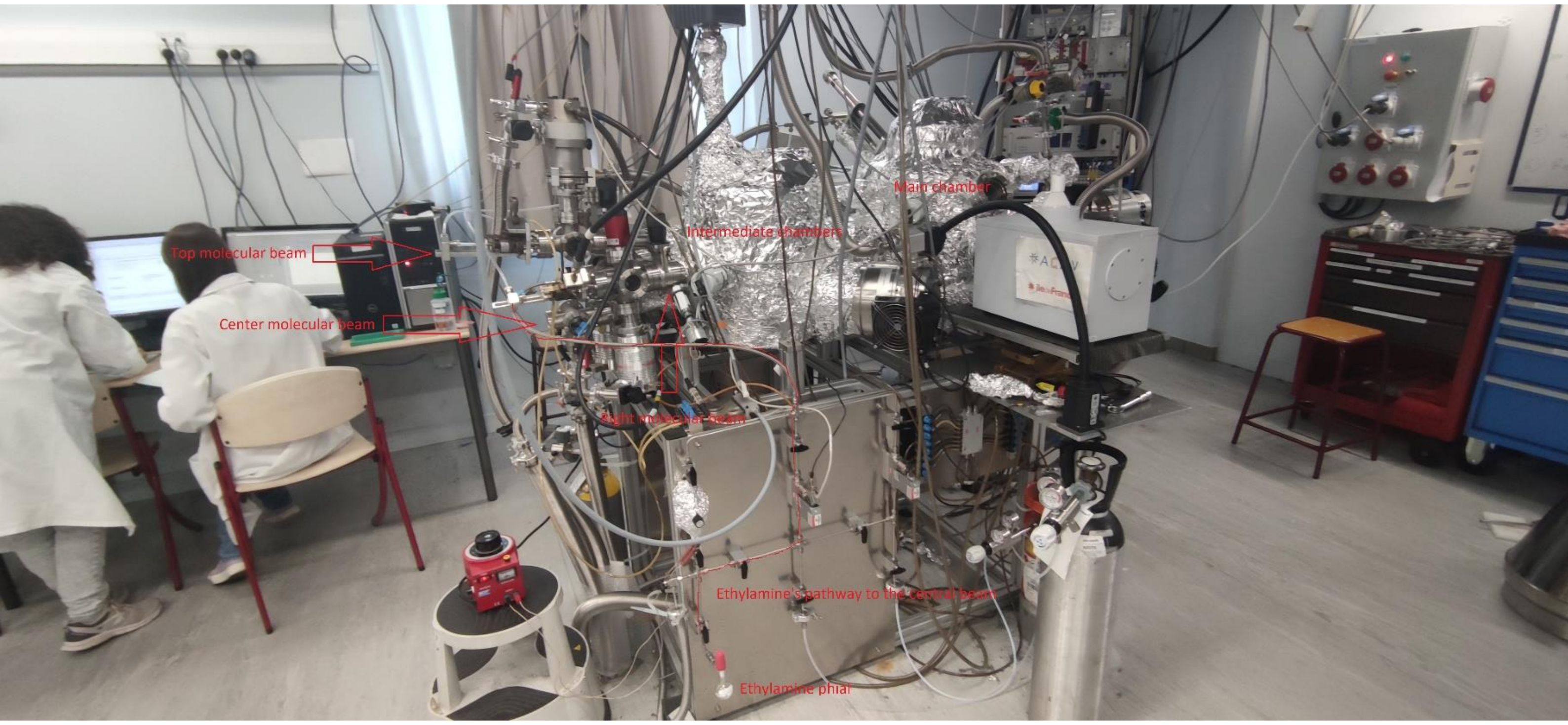
Annexes

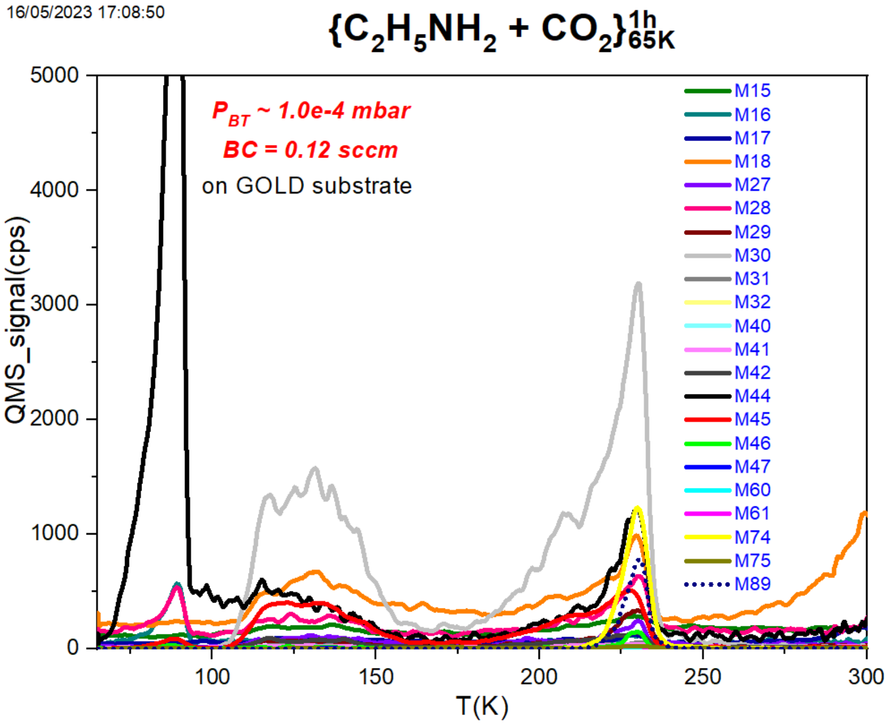
Annex 1: Picture of the VENUS apparatus



Annex 2: Pressure control screen, in mbar, BC, BT, BR stand respectively for Beam Centre, Beam Top, Beam Right, ChP stands for Chambre Principale

BC-F	4,811E-2	ChP	1,551E-9	Ch1	2,080E-7
Manifold	2,311E-2	Ch2	9,098E-9	BT	7,354E-7
InfraRoug	2,675E-1	BC	3,887E-4	BR	2,000E-2
BT-F	1,361E-2	BR-F	5,510E-2	Roots	4,996E-9

Annex 3: TPD plot corresponding to the desorbing molecules after a codeposition of ethylamine, carbon dioxide during 1 hour at 65K. All masses scanned by the QMS are shown



Annex 4: Calibration of the beams

Before each experiment campaign, the beams need to be calibrated for us to know how many molecules are sent in the chamber in a given time window. In our case, the quantity of molecules is measured in MonoLayers (ML). One monolayer is the quantity of adsorption sites on the gold surface in the area covered by the beam. It amounts[4] to about 7.10^{13} molecules in our case.

To calibrate a beam, it is set to a given flux, either through an automatic regulator or by controlling the pressure of the beam through a needle valve for example, and the molecules are deposited at low temperature (typically 10K) during a decided time. The QMS is put in front of the gold surface, and the surface is gradually heated, which leads to desorption analysed by the QMS.

The experiment is repeated several times with different deposition times, and the desorption curves are plotted together. With the increase in the doses deposited on the surface, the leading edge of the TPD curves gradually shift towards lower temperatures[11]. In fact, as the surface coverage increases, the molecules are adsorbed in less tightly bound adsorption sites. Therefore, the temperature at which the desorption starts decreases because the binding energy decreases. When the leading edge of the TPD curves stops shifting towards lower temperatures, it means that all the adsorption sites on the surface are occupied, and any other incoming molecule is adsorbed on top of the first layer of molecules already adsorbed on the surface.

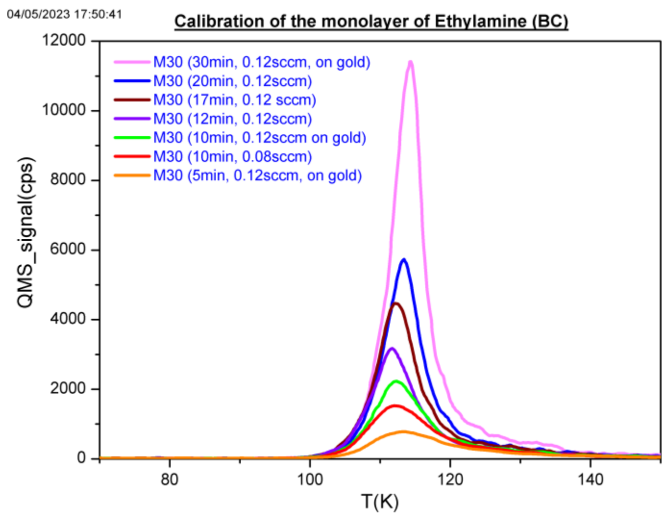


Figure 8: TPD family for $C_2H_5NH_2$ with varying deposition time and flux

Figure 8 illustrates TPD plots for ethylamine allow us to conclude that 1ML is obtained in 12 minutes with a flux of 0.12 sccm.

Some molecules, such as CO_2 show different behaviours. Specifically, CO_2 has a multilayer behaviour from the start, making it impossible to calibrate by this method. In this case, they are calibrated with the help of the IR spectra. For a given deposition time, the area of an IR band is integrated, and knowing the band strength (found in literature) the number of monolayers can be determined by the following formula[4]:

$$N_X = \frac{0.14}{0.07} \frac{\int A(\lambda)\mathrm{d}\lambda}{S_X} = 2 \frac{\int A(\lambda)\mathrm{d}\lambda}{S_X}$$

The 2 factor is an experimental factor as it takes into account the area covered by the beam as well as the density of adsorption sites.

For example, 13 minutes of deposition of CO_2 gives us an area of 0,06147 Absorbance.cm^{-1} for the main CO_2 band, and literature gives us a band strength[12] of 1.18 Absorbance.cm^{-1}.molecule^{-1}. Applying the previous formula to this data gives us a result of 1.04. Therefore, 1ML of CO^2 is obtained in 13min when deposited with a beam pressure of 1.0.10^{-4}mbar.

Annex 5: Use of IR to look for reactivity at deposition temperature

For experiments realised without water, we followed the growth of the IR CO_2 peak at 2300cm^{-1}, and compared it to CO_2 deposited alone, to see if the reaction occurred during the deposition. A reaction occurring during the deposition would consume CO_2, therefore reduce the area under the peak. Some experiments were codeposition, and some were successive deposition.

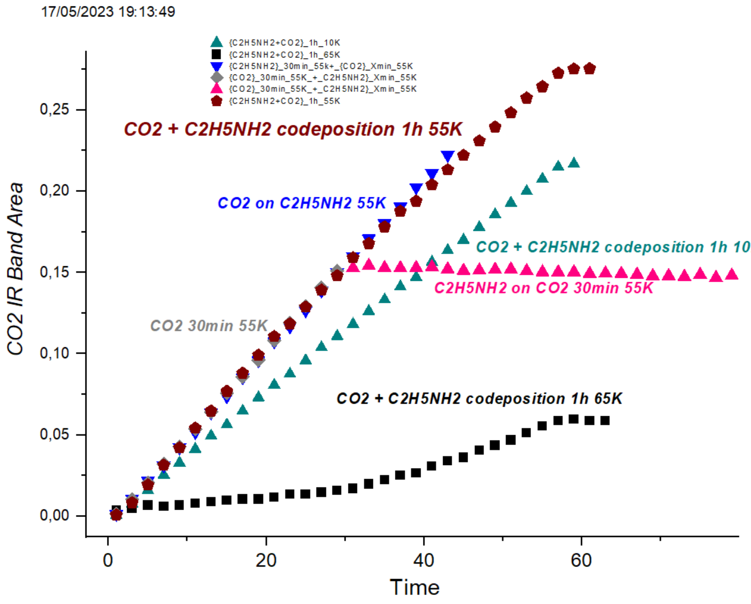


Figure 9: CO_2 band integrated area during deposition for several experiments

The experiments at 55K look like there is no reactivity at this temperature: during the codeposition, as well as when CO_2 is added onto ethylamine, the curves are the exact same than for CO_2 alone. Since our TPD shows there is product formed, it means that the reaction occurs during the TPD when the temperature starts rising. When ethylamine is deposited on 30minutes of CO_2, the amount of CO_2 very slowly decays, which could be attributed to a little bit of reactivity, or to CO_2 very slowly desorbing at 55K. The curve for the codeposition at 10K shows less CO_2 growing on the surface, which indicates that there is reactivity at 10K. This fits with our QMS data, that shows more detection of our unknown molecule when the deposition is done at 10K than when the deposition is done at 55K. Finally, the curve at 65K has CO_2 growing a lot less, which could be both a higher reactivity as well as CO_2 not sticking to the surface at this temperature. Realistically, it is a mix of both, as 65K is the temperature of deposition at which we have the highest amount of reactivity, but likely not enough to make up for the difference of CO_2 deposited on the surface.