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# Vacuum ultraviolet photoabsorption spectra of an *in-situ* synthesized peptide precursor – hydroxylamine on a cold astrochemical dust analogue

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## *Abstract*

The recent discovery of hydroxylamine (NH<sub>2</sub>OH) molecule in the interstellar medium emphasizes the need to study the molecule in laboratory astrochemical ice analogues. Here we present the first vacuum ultraviolet photoabsorption spectrum of hydroxylamine ice synthesized *in-situ* in an ammonia-oxygen (6:1) ice mixture irradiated by ~ 10 eV photons from synchrotron radiation source. The appearance of a new band between the 180 – 240 nm, with a band centered at ~207 nm, is assigned to the characteristic absorption of hydroxylamine molecule in the ice phase. This band maybe used in ammonia dominated, oxygen bearing, icy surfaces for the identification of hydroxylamine.

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## 1. Introduction

Since the detection of ammonia ( $\text{NH}_3$ ) in the Interstellar Medium (ISM) in the 1960's [1],  $-\text{NH}_2$  and  $-\text{NH}$  bearing molecules were expected to be present in the ISM. Similarly, in the outer Solar System,  $\text{NH}_3$  is a surface ice component in several icy bodies, including comets [2]. The recent discovery of ammoniated salts on comet [3] has revealed the extent of chemistry involving  $\text{NH}_3$ . Further, the detections of a host of  $\text{NH}$  bearing molecules like formamide ( $\text{NH}_2\text{CHO}$ ) [4], methanimine ( $\text{CH}_2\text{NH}$ ) [5], methylamine ( $\text{CH}_3\text{NH}_2$ ) [6, 7] and cyanamide ( $\text{NH}_2\text{CN}$ ) [8] as well as the imidogen ( $\text{NH}$ ) [9] and amino radicals ( $\text{NH}_2$ ) [10] has revealed the rich chemistry of amides and amines in the ISM.

Identification of acetamide ( $\text{CH}_3\text{CONH}_2$ ) [11], ketenimine ( $\text{CH}_2\text{CNH}$ ) [12], amino acetonitrile ( $\text{NCCH}_2\text{NH}_2$ ) [13], carbodiimide ( $\text{HNCNH}$ ) [14] and ethanimine ( $\text{CH}_3\text{CHNH}$ ) [15], N-methylformamide ( $\text{CH}_3\text{NHCHO}$ ) and even urea ( $(\text{NH}_2)_2\text{CO}$ ) [16-18] has led to an increased number of observational campaigns to unambiguously identify amino acids. Search for the presence of amino acids, the building blocks of proteins, are central to understanding the origin of life in astrobiological point of view. Glycine ( $\text{NH}_2\text{CH}_2\text{COOH}$ ), the simplest amino acid has been searched for since the 1980s [2, 19] (and references therein) and even though several detections were reported, none of it could be confirmed and the unambiguous detection of glycine is still awaited. But glycine was found in comets 67P and 82P [20, 21].

The finding of such diverse molecular derivatives of  $\text{NH}_3$  supports the argument, derived from laboratory analogue experiments, for the likely presence of peptide precursors such as hydroxylamine ( $\text{NH}_2\text{OH}$ ) on the cold dust in the ISM and radiation processed surface ices in the Solar System. Indeed, laboratory simulation of  $\text{NH}_3$  bearing ices, in ice mixtures with oxygen ( $\text{O}_2$ ) ([22] and references therein) have revealed the mechanism involved in the synthesis of  $\text{NH}_2\text{OH}$ , which is the most commonly synthesized molecule the in  $\text{NH}_3$ - $\text{O}_2$  ice mixture on a dust surface even at higher temperatures ( $\sim 200$  K). As predicted by the laboratory analogue the first detection of  $\text{NH}_2\text{OH}$  in  $\text{NH}_3$  dominated and  $\text{O}_2$  bearing regions of the ISM, has very recently been reported [23] in quiescent molecular cloud in the Galactic Center. However, Rivilla et al stressed the need for more laboratory analogue experiments to understand the formation mechanisms of  $\text{NH}_2\text{OH}$  in the ice vs gas phase.

Furthermore, the spectral signatures required to make an unambiguous identification of  $\text{NH}_2\text{OH}$  are also lacking, especially to support space based observations in the Vacuum UltraViolet (VUV) region. Here we report the first VUV photoabsorption spectrum of solid  $\text{NH}_2\text{OH}$ , synthesized in-situ in an  $\text{NH}_3$ - $\text{O}_2$  ice mixture, which will both aid our search for these molecules in icy mantles and aid our understanding on the physico-chemical nature of  $\text{NH}_2\text{OH}$  molecule.  $\text{NH}_2\text{OH}$  is a possible precursor for formation of amino acids,

therefore, our understanding of  $\text{NH}_2\text{OH}$  molecule in the ice phase will have implications in the complex biomolecules synthesis on cold ISM dust.

## 2. Experimental methodology

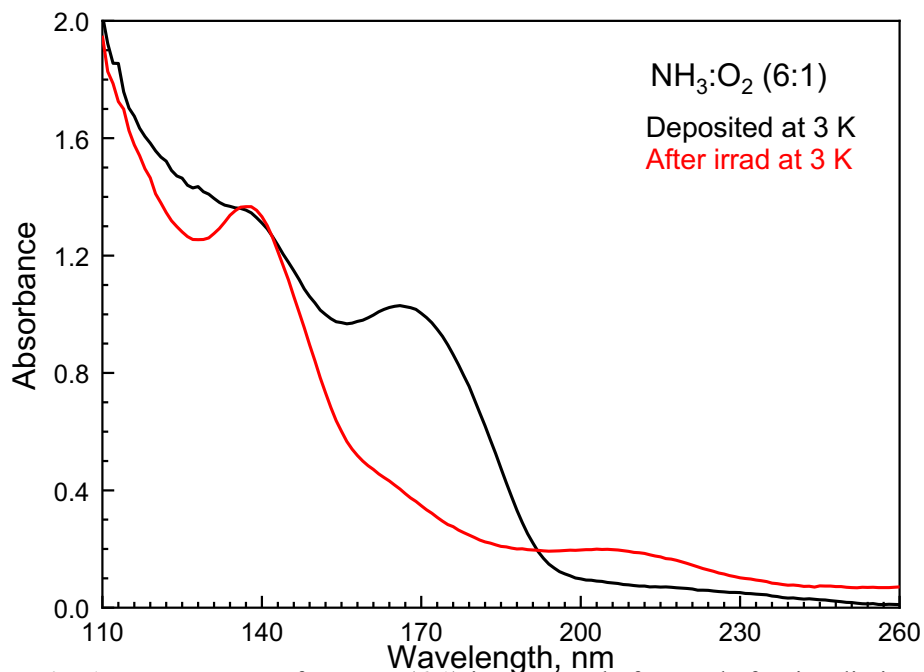
The present experiments were carried out in an end station attached to the high flux beam line BL03A1 of the storage ring (1.5 GeV) at the National Synchrotron Radiation Research Centre, NSRRC, in Taiwan. The end station is an ultrahigh vacuum chamber, pressure at  $10^{-9}$  mbar, that houses the tip of a cold head that reaches temperatures as low as 3 K. VUV transparent windows made of Lithium Fluoride (LiF) were used as the cold dust analogue on which to form molecular ices. The details of experimental setup used at the end station have been discussed earlier [24, 25].

The spectrum recorded after the LiF substrate was cooled to 3 K, and before sample deposition, was taken as  $I_0$ . After recording  $I_0$ , premixed gaseous samples of pure  $\text{NH}_3$  and  $\text{O}_2$  (6:1 ratio) were deposited, at  $5 \times 10^{-8}$  mbar for 390 seconds, approximately 20 monolayers, onto a LiF window maintained at 3 K. The spectra recorded post deposition were taken as  $I$ . Using the Beer-Lambert law VUV photoabsorption spectra of the molecular ices were derived. The photoabsorption spectrum obtained at 3 K, after deposition, was then used as a reference spectrum. The  $\text{NH}_3:\text{O}_2$  ice mixture held at 3 K was then irradiated at  $\sim 10$  eV (123.9 nm) for 3 hours and the spectra was recorded once more. The sample was then warmed (at the rate of  $5 \text{ K min}^{-1}$ ) to higher temperatures until full sublimation of the irradiated ice, with spectra being recorded at regular temperature intervals.

## 3. Results and discussion

Spectra recorded after depositing the desired ratio of  $\text{NH}_3:\text{O}_2$  mixture at on to the LiF substrate held at 3 K, showed characteristic bands corresponding to  $\text{NH}_3$  at  $\sim 170$  nm with an absorption edge around 200 nm [25, 26]. The other peak observed at  $\sim 140$  nm is assigned to the oxygen molecule in the  $\text{NH}_3$  ice before irradiation. No absorption bands were observed beyond 200 nm in the absorption spectrum after deposition and before irradiation. Upon irradiating the ice using quasi-monoenergetic ( $\sim 10$  eV) photons for three hours and recording a spectrum at the end of irradiation the intensity of the characteristic  $\text{NH}_3$  band at 170 nm reduced (Fig. 1), whilst the intensity of the 140 nm band remained broadly the same. The appearance

of a new absorption band beyond 200 nm (Fig. 1) is a clear indication of a new molecule being formed during irradiation.



**Fig. 1:** VUV spectra of NH<sub>3</sub>:O<sub>2</sub> (6:1) ices at 3 K before and after irradiation.

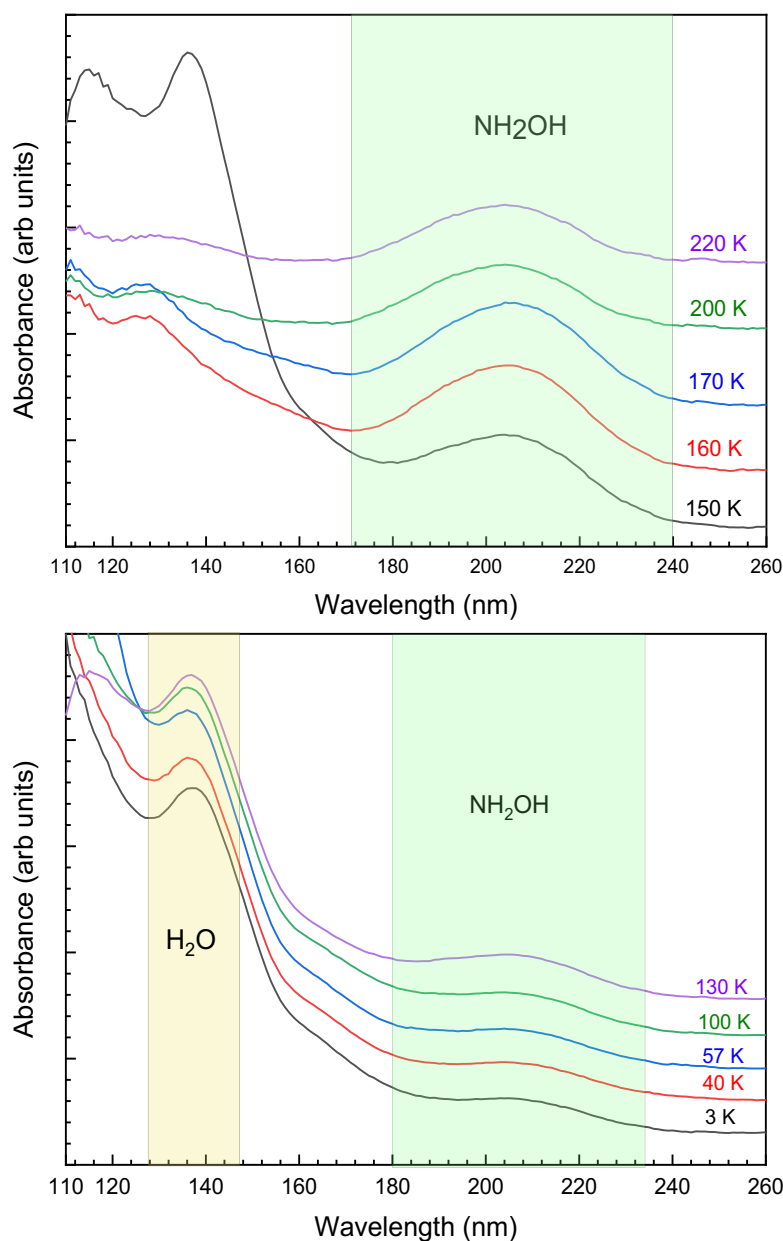
The temperature dependent spectra of the irradiated ice can reveal the chemical nature of the observed new band beyond 200 nm, as most of the NH<sub>3</sub>-O<sub>2</sub> left over from irradiation sublimates while warming the ice; less than 35 K for O<sub>2</sub> ice and less than 85 K for NH<sub>3</sub> ice. So the spectral signatures observed beyond the sublimation temperatures of these molecules we started with are clear signatures of the irradiated products. Hence, the temperature dependent spectra, of the ice post-irradiation, in comparison with studies carried out in similar ices probed using InfraRed (IR) spectroscopy [22], can be used when assigning the new peaks. By warming the ice to higher temperatures at the rate of 5 K min<sup>-1</sup> and recording spectra at different temperatures we could observe the spectrum is unaltered until 130 K, with the two prominent bands centered at 140 nm and 207 nm remaining unchanged (Fig. 2).

However, by further increasing the temperature to 150 K, the spectral signatures at 140 nm and 207 nm band were observed to more prominent, and at 160 K the 140 nm band was absent whilst the 207 nm band remained and a band centered at ~127 nm was also observed (Fig. 2). The 207 nm band remained in the spectra until 220 K, suggesting another molecule (among the products obtained from irradiation) to be responsible for this absorption at these wavelengths. This suggest that the molecule synthesized by

irradiation was sublimating at these higher temperatures and also the irradiation products must be molecules containing N, O and H atoms, free of C atoms, as we do not involve any molecule containing C atoms in the ice mixture.

Upon irradiation of NH<sub>3</sub>-O<sub>2</sub> ice N/H/O atoms are formed for reaction with the molecules in the surrounding ice. Perhaps the excess amount of NH<sub>3</sub> used in this experiments, after reacting with the O (<sup>1</sup>D) atoms, released from oxygen molecule dissociation, will readily form NH<sub>2</sub>OH [22]. There are a number of other molecules such as OH, H<sub>2</sub>O, H<sub>2</sub>O<sub>2</sub>, O<sub>3</sub>, NO, NO<sub>2</sub>, N<sub>2</sub>O, N<sub>2</sub>O<sub>3</sub> that may be to be co-synthesized in an irradiated NH<sub>3</sub>-O<sub>2</sub> ice analogue [22]. However, the abundances of such synthesized molecules are reported [22] to vary and also the sublimation temperatures of most of the molecules, listed above, lies less than or equal to 150 K. Though the sublimation temperature for molecules such as H<sub>2</sub>O<sub>2</sub> and NH<sub>2</sub>OH are known to be greater than 150 K, their relative abundances reported, (NH<sub>2</sub>OH/H<sub>2</sub>O<sub>2</sub> = 10<sup>3</sup>) [22], indicates NH<sub>2</sub>OH to be likely candidate left over on the substrate at temperatures greater than 150 K and contributing to the photoabsorption spectra at such higher temperatures. So the broad band observed at ~207 nm and the weak absorption at ~127 nm, in the spectra from 160 K to 220 K, are assigned to the NH<sub>2</sub>OH molecules (Fig. 2). In fact, the absorption band centered at ~207 nm was observed to be present in the irradiated ice at 3 K until sublimation at 220 K.

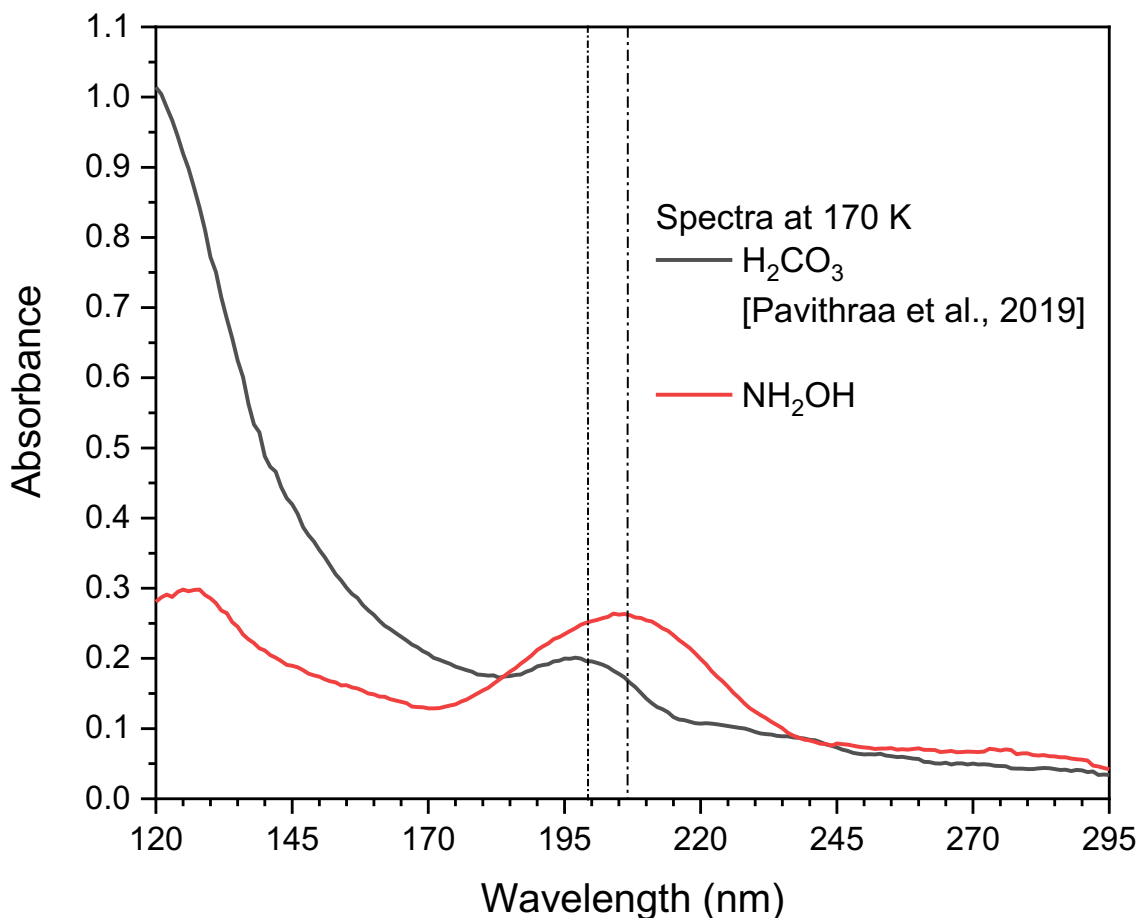
At the end of the irradiation period the significant reduction observed in the 170 nm band indicates the loss of NH<sub>3</sub> from the ice and a similar drop in intensity is expected for the 140 nm that was assigned to be from the absorption of oxygen molecule before irradiation. However, the 140 nm band remained prominent even after irradiation, suggesting the appearance of another molecule with strong absorption at 140 nm. Though oxygen molecules are dissociated releasing O atoms for reactions these can further combine with molecular oxygen synthesizing ozone, but the characteristic Hartley (240 nm – 280 nm) absorption for ozone was absent, suggesting insufficient O atoms for synthesizing ozone in sufficient concentrations to be measured. Nevertheless, ozone absorption in the VUV wavelength region has a band at 140 nm [27] even though the Hartley band was absent at higher temperatures, greater than or equal to 60 K. However, the presence of the band centered at 140 nm until 150 K without any significant change in intensity rules out the role of ozone molecule being responsible for this absorption because of its lower sublimation temperature [27].



**Fig. 2:** Temperature dependent VUV spectra of the irradiated ice warmed at the rate of 5 K min<sup>-1</sup>. Spectra are stacked for clarity.

Temperature dependent spectra revealed the 140 nm band to be prominent until 150 K but it was absent in the spectrum recorded at 160 K. Therefore, based on the nature of the irradiated ice, characteristic absorption at 140 nm [28] and sublimation temperature, we conclude H<sub>2</sub>O to be one of the major molecular component contributing to the photoabsorption spectra along with the NH<sub>2</sub>OH up until 160 K, beyond which the spectrum recorded corresponds to the NH<sub>2</sub>OH molecular ice left over from irradiation. The gas

phase spectrum of  $\text{NH}_2\text{OH}$  [29] shows strong absorption at lower wavelengths (below 160 nm) and a continuous absorption, with peak around 182 nm, until 220 nm. Whereas the VUV photoabsorption of solid phase  $\text{NH}_2\text{OH}$  shows strong absorption in the 180 nm – 240 nm with a peak around  $\sim 207$  nm.



**Fig. 3:** VUV photoabsorption spectra of in-situ synthesized pure carbonic acid and hydroxylamine at 170 K. Dotted lines indicate the peak centers.

The broad absorption band of  $\text{NH}_2\text{OH}$  was compared, in Fig. 3, with a similar band observed from the *in-situ* synthesis of carbonic acid ( $\text{H}_2\text{CO}_3$ ) molecule [30]. Both of these molecules were observed to have absorption bands lying close to one another at higher wavelengths. In fact, there is  $\sim 7$  nm shift in the band centers which is very useful for identifying these molecules. In addition,  $\text{NH}_2\text{OH}$  and  $\text{H}_2\text{CO}_3$  molecules that are synthesized *in-situ*, from VUV photon irradiation, originate from different ice mixtures;  $\text{NH}_3:\text{O}_2$  and  $\text{H}_2\text{O}:\text{CO}_2$ , respectively. Therefore, the abundance of reactant molecules, such as  $\text{NH}_3 / \text{O}_2 / \text{H}_2\text{O} / \text{CO}_2$ , on the surfaces of icy satellites or other cold surface ice bearing planetary bodies, along with the observation



of characteristic absorption bands will help us to identify the presence of such intermediate ammoniated species that are imperative for the complex molecules of life.

#### **4. Conclusion**

*In-situ* synthesized hydroxylamine in a mixture of  $\text{NH}_3\text{-O}_2$  ices subjected to 10 eV photon irradiation was found to remain on the dust analogue until 220 K with characteristic spectral signatures at VUV wavelengths. The broad band observed with a peak center at  $\sim 207$  nm, corresponding to  $\text{NH}_2\text{OH}$ , remained unaltered from lower to higher temperatures, until sublimation of the ice. As this broad band appears away from the absorption of the reactant molecules ( $\text{NH}_3 / \text{O}_2$ ), as well as from the other abundant simple molecular ices such as  $\text{H}_2\text{O} / \text{CO}_2$ , this band can be used to detect the presence of  $\text{NH}_2\text{OH}$  on the icy surfaces in the Solar System.

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#### **Author Contribution**

R Thombre: Investigation, Formal analysis, Visualization, Writing – Review & Editing

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S Pavithraa: Formal analysis, Methodology, Writing – Original draft

J–I Lo: Investigation, Resources, Formal analysis

S–L Chou: Formal analysis, Writing – Original draft

Y–J Wu: Investigation, Formal analysis, Writing – Review & Editing

R Ramachandran: Investigation, Formal analysis, Visualization, Writing – Review & Editing

K K Rahul: Formal analysis

B -M Cheng: Investigation, Resources, Formal analysis, Writing – Review & Editing

H Hill: Formal analysis, Investigation, Writing – Review & Editing

A Bhardwaj: Formal analysis, Investigation, Resources, Writing – Review & Editing

B N Rajasekhar: Formal analysis, Investigation, Visualization, Writing – Review & Editing

N J Mason: Formal analysis, Resources, Visualization, Funding acquisition, Writing – Review & Editing

B Sivaraman: Conceptualization, Supervision, Project administration, Methodology, Investigation, Formal analysis, Visualization, Funding acquisition, Writing – Original draft

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