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Refractive index and density of ammonia ice at different temperatures of deposition

M. Á. Satorre^{*a}, J. Leliwa-Kopystynski^b, C. Santonja^a, R. Luna^a

^a*Centro de tecnologías Físicas. Universitat Politècnica de València, 46022 Valencia, Spain*

^b*Warsaw University, Institute of Geophysics, Pasteura 7, 02-093 Warszawa, Poland*

Abstract

Despite its relevance in astrophysical scenarios, optical properties and density of ammonia have been scarcely studied. This work presents new data on the real part of the refractive index of ammonia at 632.8 nm and density at different temperatures of deposition from 13 K up to its desorption temperature around 110 K. The results show a significant variability for both parameters versus temperature, representing an increase of 50 % for density and a 10 % for refractive index as temperature increases in the range 13 - 60 K, and at temperatures higher than 60 K a constant value is reached for both parameters. This initial variation and the further plateau reflects structural differences in its form at low and high temperatures that can play an important role in its interaction with other molecules of astrophysical interest.

Keywords:

Ices, Experimental techniques, Satellites surfaces, Trans-neptunian objects, Comets

1. Introduction

Ammonia ice is present in many astrophysical scenarios in Solar System bodies, on satellites as Enceladus (Emery et al., 2005; Verbiscer et al., 2006), Miranda (Bauer et al., 2002), on TNO's as Charon (Brown & Calvin, 2000; Dumas et al., 2001; Cook et al., 2006, 2007), or Quaoar (Jewitt & Luu, 2004), even in comets (Kawakita & Watanabe (2002) and references therein). All these works confirm the prediction of Lewis (1972) who proposed ammonia as one of the most abundant molecules incorporated into the outer Solar System bodies, recently Pizzarello & Williams (2012) confirmed the presence of ammonia from carbonaceous carbonites in the Early Solar System. Moreover ammonia modifies water properties, (Kargel, 1992; Lodders, 2003; Marion et al., 2012), even could be important not only for the surfaces but even for inner parts of icy satellites (Leliwa-Kopystynski et al., 2002). However, scarce studies have been performed on

the optical properties of this ice under astrophysical conditions (low pressure and low temperature). These studies are needed in a wide range of applications in planetary science and astrophysics to improve the understanding on the properties of these scenarios.

The theory of thin film optics has been described extensively in the literature (Born and Wolf, 1999; Heavens, 1991) and several techniques have been used to measure (in different spectral regions), the real part of the refractive index (hereafter, n) (Tempelmeier and Mills, 1968).

Concerning ammonia, Romanescu et al.(2010) obtained a refractive index almost constant around 1.49 at three different temperatures of 80, 90 and 100 K. Other authors encountered some values at low temperatures (20-30 K) in the range 1.37-1.43 (Pipes et al., 1978; Wood & Roux, 1982; Dawes et al., 2007), however, despite its relevance, no results for ammonia ice have been reported below 20 K and in the interval from 30 K up to 75 K (as far as we know). The main reason is probably its high reactivity that hampers its management in the laboratory.

Concerning density (hereafter ρ), experimental

*Corresponding author. Tel: +34966528542

Email address: msatorre@fis.upv.es (M. Á. Satorre*)

46 data are even more scarce, since the few reported 98
 47 data are obtained at around 80 K or around 25 99
 48 K. The results published so far show higher val- 100
 49 ues when density is obtained at 80 K. These results 101
 50 obey the same behaviour than n i.e.: significantly 102
 51 lower values of density at low temperatures (20 K) 103
 52 (Wood & Roux, 1982) respect that obtained around 104
 53 80 K (Olovsson & Templeton, 1959; Manzhelii & 105
 54 Tolkachev, 1964; Wood & Roux, 1982).

55 Differences in both, n and ρ , at low and high 106
 56 temperatures invite to infer different structures. 107
 57 During decades authors agree about an amorphous 108
 58 structure at low temperatures and a crystalline one 109
 59 at high temperatures. But no agreement is achieved 110
 60 about a likely metastable structure that seems to 111
 61 appear in a different range of temperatures depend- 112
 62 ing on the authors. To clarify this controversial 113
 63 question, two different experimental works (Zheng 114
 64 & Kaiser, 2007; Dawes et al., 2007) has been re- 115
 65 cently performed and both conclude in their results 116
 66 the existence of only two different structures: an 117
 67 amorphous one below around 60 K (hereafter low 118
 68 temperatures means below 60 K) and a crystalline 119
 69 one for temperatures higher than 60 K (hereafter 120
 70 high temperatures means above 60 K).

71 To help to disentangle the problem of the differ- 121
 72 ent structures of ammonia (amorphous, metastable 122
 73 and crystalline phase), it would be relevant finding 123
 74 additional values of n and ρ for different tempera- 124
 75 tures of those present in the literature. 125

76 On the other hand n and ρ are relevant by them- 126
 77 selves for different. If the refractive index of a 127
 78 species is known, it is possible to calculate the thick- 128
 79 ness of a deposited film from the number of interfer- 129
 80 ence fringes obtained with a coherent light beam. In 130
 81 our experiments we determine n at 632.8 nm (He- 131
 82 Ne laser). This data is especially useful because the 132
 83 He-Ne wavelength is commonly used as a standard 133
 84 in almost any laboratory. Thickness is needed in 134
 85 the determination of another very important phys- 135
 86 ical characteristic as density, which is relevant in 136
 87 the assessment of many magnitudes of astrophys- 137
 88 ical interest as the integrated absorbance strength, 138
 89 porosity, penetration depth of ions impinging an 139
 90 ice, etc. 140

91 In the case of experiments of irradiation, the pen- 141
 92 etration depths depends directly on density and 142
 93 determine whether an experiment is a thin (ions 143
 94 passing the film) or thick (ions stopped in the film) 144
 95 film experiment, by determining the stopping power 145
 96 with specialized programs as Ziegler's SRIM pro- 146
 97 gram (Ziegler et al. (1985)). 147

Density is also important in the analysis of many
 results: observational, experimental and theoret-
 ical. The integrated absorbance value A (cm
 molecule⁻¹), can be used to estimate column densi-
 ties of ice constituents from the absorption spectra
 of the ices (Hudgins et al., 1993). Integrated ab-
 sorbance is defined as:

$$A = \frac{1}{Cl} \int_{\nu(1)}^{\nu(2)} \tau_{\nu} d\nu$$

where C is the concentration of the absorbers,
 often expressed as molecule cm⁻³ (or mol l⁻¹), l is
 the path-length of the beam through the material, ν
 is the frequency in cm⁻¹, $\tau_{\nu} = \ln(I_0/I)$ is the ratio
 of the incident to the transmitted beam, with the
 integration performed over the band. C is obtained
 as follows:

$$C = \frac{\rho N_A}{Mr}$$

where ρ is the density of the ice in g cm⁻³, N_A is
 the Avogadro constant in molecule mol⁻¹ and Mr
 is the molecular mass in g mol⁻¹. d'Hendecourt
 & Allamandola (1986) assumed that, for the cal-
 culation of A , the largest uncertainties arises from
 evaluating C (concentration of absorbers), as the
 densities of ices are not accurately known. They
 assumed the densities to be constant (1 g cm⁻³).
 This assumption was also made by many other au-
 thors, among them is used by Hudgins et al. (1993)
 in their exhaustive work to determine optical con-
 stants and integrated absorbance for mid- and far-
 infrared spectroscopy of ices.

Density is even relevant by itself in the physical
 chemistry of ices. The course of chemical differen-
 tiation depends very much on the relative densities
 of important phases and whether they tend to sink
 or float (Kargel, 1991). This author presents densi-
 ties of representative cryovolcanic liquids and solids
 versus temperature. Knowing the density at tem-
 peratures relevant for astrophysical scenarios helps
 to understand part of their surface dynamics.

We present in this work new results on the real
 part of refractive index at 632.8 nm and bulk den-
 sity of NH₃ at different temperatures (ranging from
 10 to 100 K). These results can be used to bet-
 ter reproduce irradiation, integrated absorbances
 and buoyancy of ices at different temperatures and
 could help to better understand the structure of
 ammonia. In this article, a review of the exper-
 imental setup and procedures is described in the
 next section. In section 3 the experimental results
 are showed and discussed, and finally in section 4
 the conclusions are exposed.

148 **2. Experimental**

149 The experimental apparatus and the procedure
 150 to obtain the refractive index and density is briefly
 151 summarized here. A more detailed explanation in
 152 some aspects is presented in Satorre et al. (2008).
 153 A simple schematic of the apparatus is shown in
 154 Figure 1.

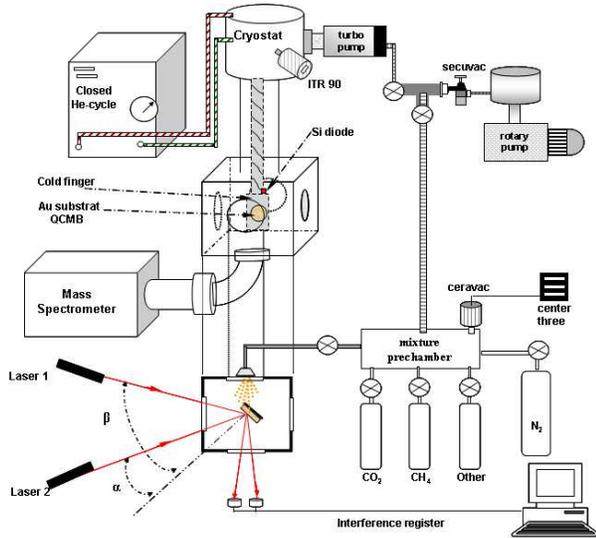


Figure 1: Experimental setup

155 We have measured refractive index and density of
 156 NH_3 ice under high vacuum conditions (base pres-
 157 sure $< 10^{-7}$ mbar) and temperature ranging from
 158 13 to 100 K.

159 Sample holder (quartz crystal microbalance,
 160 hereafter QCMB) temperature is operated by the
 161 Intelligent Temperature Controller ITC 503S (Ox-
 162 ford Instruments). It uses the feedback of a silicon
 163 diode sensor (Scientific Instruments) located just
 164 beside the quartz, that allows the temperature to
 165 vary between 10.0 and 300.0 ± 0.5 K, by means a
 166 resistive heater.

167 Refractive index has been obtained by double
 168 laser interferometry at 632.8 nm. This technique
 169 has been widely used in the literature as for exam-
 170 ple in the work of Tempelmeyer and Mills (1968).

171 The QCMB is used to calculate the mass of ice
 172 accreted per unit area (in g cm^{-2}). This value is
 173 obtained from the QCMB variation in frequency by
 174 using the Sauerbrey equation: $\Delta f = -S \cdot \Delta m$. In
 175 this equation Δf is variation in frequency, Δm rep-
 176 represents the mass accreted onto the balance and S is

177 a specific constant for every QCMB. From the value
 178 obtained with the QCMB signal and the thickness,
 179 density is obtained.

During deposition, gas flows from the precham-
 180 ber to the deposit vacuum chamber controlled by
 181 a needle valve (Leybold D50968). To form the ice
 182 film, a constant rate of deposition is used (around
 183 $1 \mu\text{m h}^{-1}$), maintaining almost constant the aper-
 184 ture of the valve. NH_3 enters the deposition chamber
 185 and deposit on the coldest parts growing a film onto
 186 the QCMB. The pressure of ammonia and contam-
 187 inants composition during film growth is checked
 188 through the Quadrupole mass spectrometer (QMS)
 189 (AccuQuad RGA 100 with a resolution of ~ 0.5
 190 amu). Some experiments, at different tempera-
 191 tures, are repeated to check its reproducibility and
 192 all the results are within the error bars. Ammonia
 193 used is Praxair 99.999.

Table 1: Density (ρ) and refractive index (n), obtained for NH_3 at different temperatures of deposition compared with the literature.

T. (K)	Density (g cm^{-3})		Refractive index	
	$\rho \pm 5\%$	Lit.	$n \pm 2.5\%$	Lit.
13	0.67		1.38	
20	0.72	0.76 ^a	1.40	1.37 ^a 1.42 ^b
25				1.44 ^c
40	0.80		1.45	
60	0.89		1.49	
75				1.48 ^c
77		0.861 ^d		
80	0.87	0.87 ^a	1.49	1.41 ^a 1.49 ^e 1.42 ^b
81		0.863 ^f		
90	0.90		1.48	1.50 ^e
100	0.85		1.48	1.48 ^e 1.4 ^c

References

- a: Wood and Roux(1982)
- b: Pipes et al.(1978)
- c: Dawes et al.(2007)
- d: Olovsson and Templeton(1959)
- e: Romanescu et al.(2010)
- f: Manzhelii and Tolkachev(1964)

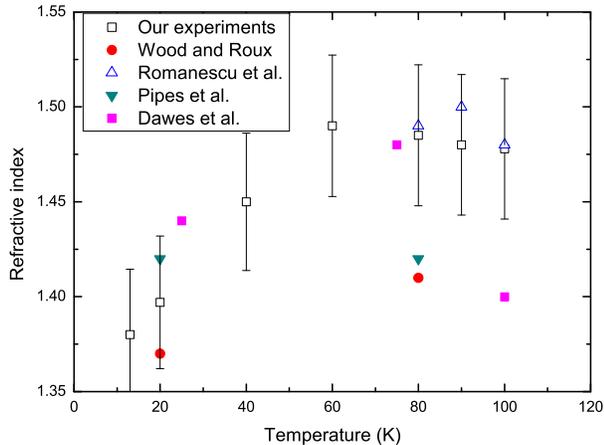


Figure 2: NH_3 refractive index at different temperatures of deposition compared with data obtained from the literature.

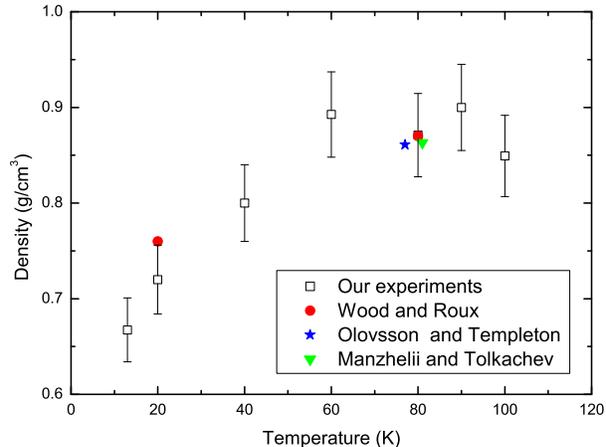


Figure 3: NH_3 density at different temperatures of deposition compared with data obtained from the literature.

3. Results and discussion

The motivation of our work is to provide new data for n and ρ at temperatures at which they are no previously obtained. These data are useful because from previous results is clear that ammonia ice structure depends on the temperature of deposition. In this section we discuss the validity of our results comparing with experimental data obtained with both, the same and other experimental techniques.

Density and refractive index obtained at different temperatures in our set of experiments (ranging 13 to 100 K), has been collected and presented in Table 1 together with other values taken from the literature, in order to compare them. Additionally, all the data for n and ρ have been plotted in Figure 2 and 3 respectively. The errors considered in these graphs are 2.5 % for n and 5 % for ρ . Errors in the temperature are not marked since they are within the symbol size.

Our experimental data show two different behaviours, below and above 60 K. Below it (left side in Figures 2 and 3), n and ρ vary with temperature as $n = (1.35 + 0.00231 T)$ and $\rho = (0.628 + 0.00462 T) \text{ g cm}^{-3}$, with n growing from 1.38 to 1.48 and ρ increasing from 0.67 to 0.87. In both cases a plateau is reached for temperatures higher than 60 K (right side in Figures 2 and 3), being $n_{\text{plateau}} = 1.48 \pm 0.05$, and $\rho_{\text{plateau}} = 0.67 \pm 0.02 \text{ g cm}^{-3}$. If we substitute, in the straight fits, the plateau values of n and ρ versus temperature is reached, we obtain a temperature for transition of $58 \pm 5 \text{ K}$. These

results suggest a progressive increase of structural order for ammonia molecules below 60 K and a stable structure above it.

These results complete those published so far and agree with most of them (Figures 2 and 3). Only deviations around 5% are observed for n at temperatures around 75 K (Figure 2). This particular discrepancy will be discussed in more detail later.

Looking at the literature data, the main conclusion is that around 25 K, values obtained for both n and ρ are lower than those obtained at around 80 K or higher. Because the lack of data below 25 K and from 30 up to 80 K it is not possible to establish a relationship between n and ρ and the structure of ammonia. Only three constant n values presented by Romanescu et al. (2010) at 70, 80 and 90 K let us deduce a stable structure.

Refractive index values are usually obtained by double laser interferometry. Dawes et al. (2007) used a technique explained in detail by Westley et al. (1998) based on the interference curves with one laser. Among all these results (including ours), those obtained by Romanescu et al. (2010) by double laser interferometry are probably the most accurate due to their number of experiments for a specific temperature (16 experiments, at 50 K) and because they analyze and subtract the output of the laser signal (using an additional sensor) correcting the artifacts of the device. This procedure presents the advantage that correcting the variation in the laser beam, the interference curves are clarified. Our results, taking into account the error bars,

259 fully agree with the results of Dawes et al. (2007) at 311
 260 77 K, and that of Romanescu et al. (2010) at 80, 90 312
 261 and 100 K (see Figure 2). Dawes et al. (2007) work 313
 262 under UHV conditions and their results at 100 K 314
 263 could be affected because under UHV conditions, 315
 264 depending on the pressure of deposition, two processes 316
 265 could compete in the dynamics of the process, 317
 266 deposition and sublimation. 318

267 Pipes et al. (1978) and Wood & Roux (1982) use 319
 268 both almost the same experimental setup. As it 320
 269 is clearly shown in Figure 2, at high temperatures 321
 270 both works obtain lower values than any other 322
 271 authors (including the result obtained in this work) 323
 272 but at 20 K their results are within the error bars
 273 of ours. The discrepancy between the results obtained
 274 by these authors at high temperatures and all the others,
 275 is explained by Romanescu et al. (2010) from the
 276 deposition rate in the experiments of Pipes et al. (1978)
 277 and Wood & Roux (1982) (in the order of $\mu\text{m min}^{-1}$).
 278

279 The explanation of the discrepancy, more evident
 280 for crystalline than for amorphous structure, could be
 281 that forming any crystalline structure, in the case of
 282 ammonia a Face Centered Cubic structure (thereafter
 283 FCC, see Figure 4) needs a certain experimental
 284 conditions. At higher temperatures than 60 K a
 285 crystalline structure is formed if deposition conditions
 286 allow it. A new molecule arriving needs a certain
 287 time to achieve the appropriate position and orientation.
 288 For almost all the experiments performed by many
 289 authors with different techniques and different rate
 290 of deposition, all of them conclude that no dependence
 291 of the rate of deposition is observed, but it seems
 292 that this is true up to a certain threshold. If it is
 293 exceeded, molecules have no time to reorganize in
 294 a crystalline structure. This threshold should be
 295 around that rate used by Pipes et al. (1978) and
 296 Wood & Roux (1982) as Romanescu et al. (2010)
 297 point out as the cause of the discrepancy between
 298 their results and those of Pipes et al. (1978),
 299 Wood & Roux (1982). If deposition rate finally
 300 influences on the result, this effect should be
 301 more relevant for the crystalline phase than for
 302 amorphous ice, this would explain why at 25 K
 303 their data agree with the other ones. 324

304 Concerning ρ values, all the results for all the
 305 temperatures present in the literature fully agree
 306 with our results (see Figure 3), that of Wood &
 307 Roux (1982), at 20 and 80 K, using the same
 308 experimental technique of us, Manzhelii & Tolkahev
 309 (1964) using picnometer, and Olovsson & Templeton
 310 (1959) using X-ray diffraction at around 80 K. 325

311 Romanescu et al. (2010) claim the high accuracy of
 312 the experimental technique using picnometers and
 313 X-ray diffraction for the ρ values. Our results are
 314 within 1% to those last works, and, despite the
 315 difficulties of working with the QCM (mentioned by
 316 Romanescu et al. (2010)).

317 Our results provide for the first time, enough
 318 additional data to show a clear linear increase in
 319 n and ρ values from low temperatures up to 60 K
 320 and additionally they confirm the presence of a
 321 steady state above 60 K. Then all the experimental
 322 results point to, at least, two different structures,
 323 below and above 60 K.

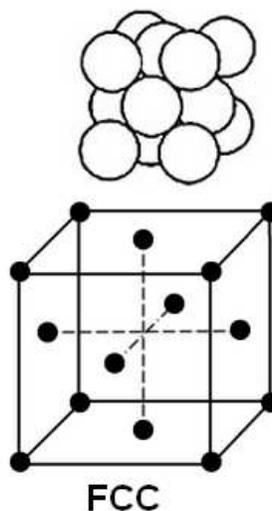


Figure 4: Crystal structure for NH_3 .

324 4. Conclusions

325 In this work the density and refractive index for
 326 NH_3 at different temperatures of deposition have
 327 been determined.

328 These values complete a lack of data at different
 329 temperatures from 13 up to 100 K. Two different
 330 behaviours are obtained, in a first range of temper-
 331 atures (from 13 to 60 K), both n and ρ increase
 332 their values linearly with a rate of 0.00231 K^{-1} for
 333 n and $0.00462 \text{ g cm}^{-3} \text{ K}^{-1}$ for ρ . Above 58 K a
 334 constant value is obtained for both n and ρ ($n =$
 335 1.48 ± 0.05 and $\rho = 0.87 \pm 0.02 \text{ g cm}^{-3}$), in ex-
 336 cellent agreement with the literature (see Figures
 337 2 and 3). Specifically our value for the transition
 338 temperature from amorphous to crystalline phase

339 obtained crossing the plateau with the straight line
340 fits is 58 ± 5 K.

341 The transition temperature deduced from our
342 procedure is almost the same than that obtained by
343 other authors (Dawes et al., 2007; Zheng & Kaiser,
344 2007).

345 Despite this agreement, taking into account our
346 experimental error is not possible to discard a
347 metastable structure that has been a controversial
348 point during a long time. This metastable structure
349 was initially proposed by Staats & Morgan (1959)
350 from the work of Reding & Horning (1951), whose
351 results has been reproduced by many authors as re-
352 vised Ferraro et al. (1980). This structure has also
353 been found by Moore and Hudson (1994) but they
354 called the range at which this features appear as
355 “transition phase”. In order to elucidate the ap-
356 pearance or not of a metastable structure, more
357 recent works of Dawes et al. (2007) and Zheng &
358 Kaiser (2007) have been designed with the aim of
359 clarifying whether this metastable phase of ammo-
360 nia exists and, if is the case, the range of tempera-
361 ture in which it is present.

362 Dawes et al. (2007) divide the dependence of
363 the structure in two ranges: below and above 65
364 K where the transition occurs from amorphous to
365 crystalline FCC. This study is based on the varia-
366 tion of the IR and Visible-UV spectrum of ammo-
367 nia deposited at 25 K and comparing it with the
368 spectra obtained at different temperatures during
369 warming up. Additionally, they obtain spectra for
370 NH_3 deposited directly at higher temperatures and
371 compare them with those deposited at low temper-
372 atures and subsequently warmed up.

373 The study of Zheng & Kaiser (2007) points out
374 the same behaviour but establishing the transition
375 point at slightly lower temperature: 58 K. Despite
376 these authors studied the sample only with the IR,
377 their higher experimental resolution respect Dawes
378 et al. (2007), consents them to show a fine struc-
379 ture of the spectra appearing at 58 K that indi-
380 cates structural changes associated to the crys-
381 talline form.

382 Zheng & Kaiser (2007) performed additional ex-
383 periments using mixtures of ammonia with water
384 ice, because water is the most important contami-
385 nant in high vacuum systems and because its high
386 reactivity with ammonia in the prechamber. They
387 found that when ammonia is mixed with 1 % of wa-
388 ter, the transition temperature changes from 58 to
389 65 K. These results show that the presence of water
390 ice even as a contaminant, could vary the transition

temperature from amorphous to crystalline.

Ice of carbon dioxide presents also a similar vari-
392 ation in temperature for n and ρ , but presenting
393 and increase with temperature higher than in the
394 case of NH_3 (Satorre et al., 2008). This lower vari-
395 ability in the case of ammonia could be associated
396 to the H-bonds present in this molecule.

Our work is relevant because ammonia is abund-
398 ant in many astrophysical scenarios. In those,
399 some additional aspects must be taken into consid-
400 eration: i) The buoyancy of mixtures when ammo-
401 nia is present in a mixture liquid-solid context, will
402 be greater than expected from their previous used
403 value as its density could decrease down to 20 % (as
404 temperature decreases) from the usually assumed
405 value of 1 g cm^{-3} . ii) Irradiation dose (Moore et
406 al., 2007) must be revised for experiments where
407 NH_3 is involved since penetration depth is affected
408 by density of the film irradiated, therefore from our
409 results ammonia could present values of penetration
410 higher than expected. iii) Abundance calculated
411 for the NH_3 (Hudgins et al., 1993) in the scenarios
412 where is present in solid state must also be revised
413 considering our values for calculation purposes.

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