

# Experimental Effervescence and Freezing Point Depression Measurements of Nitrogen in Liquid Methane-Ethane Mixtures

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**Abstract.** NASA is designing an unmanned submarine to explore the depths of the hydrocarbon-rich seas on Saturn's moon Titan. Data from Cassini indicates that the Titan north polar environment sustains stable seas of variable concentrations of ethane, methane, and nitrogen, with a surface temperature near 93 K. The submarine must operate autonomously, study atmosphere/sea exchange, interact with the seabed, hover at the surface or any depth within the sea, and be capable of tolerating variable hydrocarbon compositions. Currently, the main thermal design concern is the effect of effervescence on submarine operation, which affects the ballast system, science instruments, and propellers. Twelve effervescence measurements on various liquid methane-ethane compositions with dissolved gaseous nitrogen are thus presented from 1.5 bar to 4.5 bar at temperatures from 92 K to 96 K to simulate the conditions of the seas. After conducting effervescence measurements, two freezing point depression measurements were conducted. The freezing liquid line was depressed more than 15 K below the triple point temperatures of pure ethane (90.4 K) and pure methane (90.7 K). Experimental effervescence measurements will be used to compare directly with effervescence modeling to determine if changes are required in the design of the thermal management system as well as the propellers.

**Keywords:** Effervescence, Methane-Ethane Mixtures, Extraterrestrial Submarine, Titan, freezing point depression

## 1. Introduction

Saturn's moon Titan is the only known celestial body in our solar system besides Earth with stable liquid seas accessible on the surface (Stofan et al., 2007). NASA is currently designing an unmanned autonomous submarine to explore these methane-ethane rich seas: 1) to study the evolution of hydrocarbons in the universe, 2) to study Titan's geology (atmosphere/sea exchange, surface, shore, waves, heat transfer), and 3) to provide a pathfinder for later designs of submersibles in the seas hidden beneath the ice crust of other outer planetary moons (Hartwig et al., 2016). The submarine has the advantage of being able to conduct measurements of the atmosphere and seas.

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39 Data from Cassini indicates that the surface temperature of Titan is approximately 93 K with an  
40 atmospheric pressure of 1.5 bar (Mitri et al., 2007; Lorenz and Mitton, 2010). The seas vary in  
41 the amount of liquid methane and ethane (Lorenz et al., 2014; Tokano and Lorenz, 2016). Unlike  
42 the liquid water oceans of Earth, the hydrocarbon seas of Titan are able to absorb a relatively  
43 substantial amount of nitrogen from the atmosphere, causing gaseous nitrogen to go into  
44 solution. The solubility of nitrogen varies dramatically depending on the composition of ethane  
45 and methane in the seas, which can vary from nearly pure ethane in Kraken Mare to 74 mol %  
46 methane in Ligeia Mare (Hartwig et al., 2016). At Titan's surface conditions of 93 K and 1.5 bar,  
47 the solubility of nitrogen in Ligeia Mare is estimated to be 12-13 mol % while the solubility of  
48 nitrogen in Kraken Mare is estimated at just 3 mol % (Hartwig et al., 2017). This range in  
49 solubility presents several design challenges and creates uncertainty regarding ice formation in  
50 the seas due to Titan's surface temperature being within 2 K to 3 K of the triple point of pure  
51 methane and ethane. Though there is limited experimental data, the literature suggests that  
52 significant freezing point depression can be achieved, and that the buoyancy of the ice will  
53 depend on the solubility of nitrogen and the methane-ethane composition of the sea (e.g.  
54 Thompson, 1985; Roe and Grundy, 2012; Prokhvatilov and Yantsevich, 1983; Hofgartner and  
55 Lunine, 2013). Even though the depth of Ligeia Mare is known to be relatively shallow at 200 m  
56 (Mastrogiuseppe et al., 2014), it is unknown if the temperature gradient is enough to incite  
57 freezing at these depths.

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59 The primary thermal design concern of the submarine is predicting the effects of nitrogen  
60 effervescence on submarine operation. The 350-400 W/m<sup>2</sup> waste heat flux from the submarine  
61 radioisotope power system is not enough to boil the surrounding seas, but it may cause dissolved  
62 nitrogen gas to come out of solution (Hartwig et al., 2016). In a quiescent case, bubbles may  
63 interfere with sensitive science measurements. In a moving case, bubbles that form along the  
64 body may coalesce at the aft end of the submarine and cause cavitation in the propellers. Due to  
65 the high solubility of nitrogen in the Titan seas, data and models are needed to quantify the  
66 amount of dissolved gas, as well as conditions that will cause bubbles to form, grow, and  
67 coalesce along the submarine.

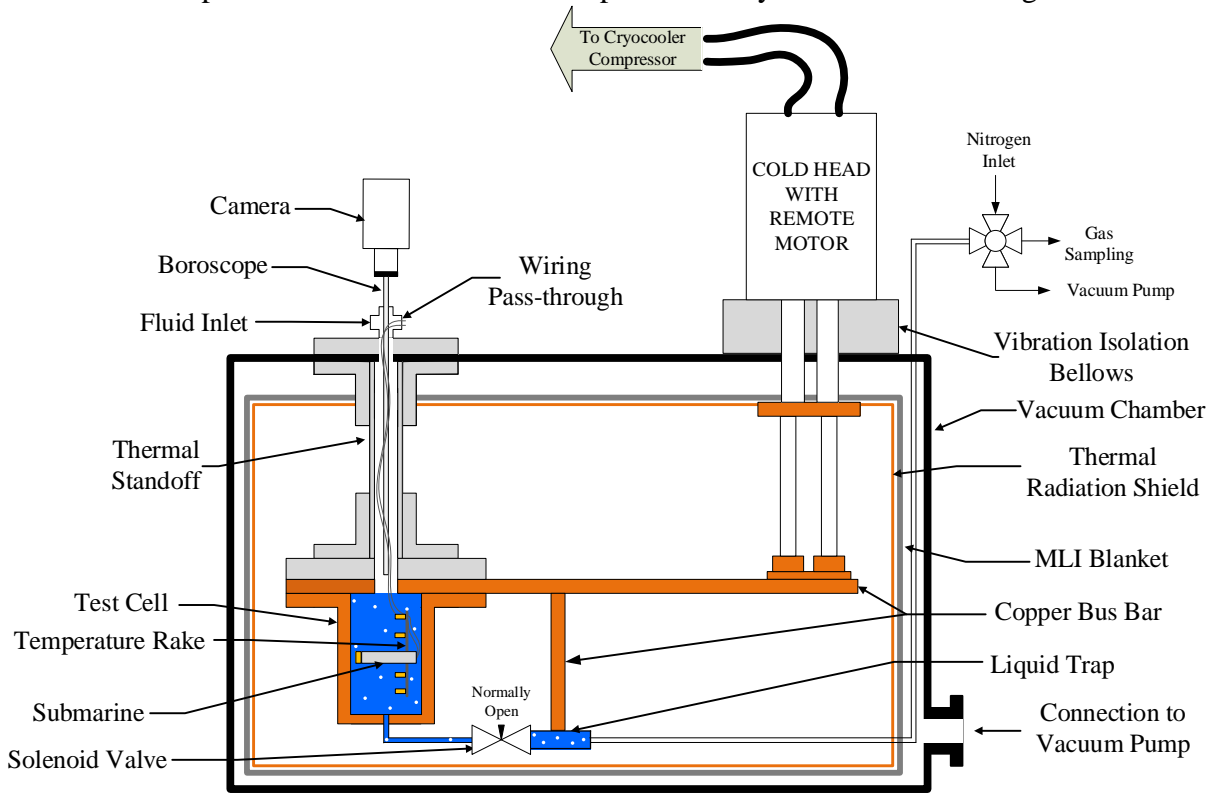
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69 Effervescence and nitrogen dissolution in the seas of Titan has been modelled and discussed  
70 previously with respect to composition change and bulk warming/cooling (Cordier et al., 2017;  
71 Malaska et al., 2016). The current work focuses specifically on the effects of heat dissipation  
72 from a Titan submarine. Experimental measurements were conducted to determine the heat  
73 fluxes and surface temperatures at which nitrogen gas begins to come out of solution to  
74 determine the point of bubble incipience as a function of sea temperature, pressure, and liquid  
75 methane-ethane compositions. Videos of effervescence were taken to better understand the  
76 impact that nitrogen gas bubbles may have on the scientific instruments and submarine  
77 propellers. Additionally, two freezing point depression measurements were conducted on  
78 methane-ethane-nitrogen mixtures to determine the degree of depression.

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## 80 **2. Experimental Design**

81 The effervescence measurements presented in this work were completed using the same cryostat,  
82 bulk gases, mixing tank, and liquid trap that were used to conduct pressure-density-temperature-  
83 composition measurements on liquid methane-ethane-nitrogen mixtures relevant to Titan

84 (Richardson et al., 2018). The key components of the experimental system include a custom  
 85 cryostat and test cell to condense methane-ethane mixtures, video camera with a borescope,  
 86 cartridge heater to simulate waste heat from the submarine, and liquid trap which is used to  
 87 determine the composition. A schematic of the experimental system is shown in Figure 1.



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 90 Figure 1. Experimental system used to conduct effervescence measurements.  
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92 Gas cylinders of 99.99 % pure ethane and 99.99 % pure methane were mixed based on partial  
 93 pressure in a mixing tank to achieve the approximate desired composition using Dalton’s law.  
 94 The gaseous mixture was then condensed via cryo-pumping in the copper test cell where  
 95 nitrogen was bubbled in through the liquid trap at the bottom until the desired total pressure was  
 96 achieved. Nitrogen bubbling was a turbulent process ensuring that the liquid mixture within the  
 97 liquid trap and test cell was well mixed. The temperature of the test cell and liquid was  
 98 controlled using a Proportional-Integral-Derivative (PID) temperature controller and electric  
 99 heater placed on the outside of the test cell. The temperature controller supplied power to the  
 100 heater to maintain a specified liquid temperature. Once the temperature and pressure of the  
 101 simulated sea was stable, the cartridge heater was turned on to simulate the heat given off by the  
 102 submarine. A summary of the sensors and instruments used to conduct the effervescence  
 103 measurements is presented in Table 1.  
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105 Table 1. Summary of instrumentation and sensors used to conduct effervescence measurements.

Measurement	Instrument	Accuracy
Pressure	Paroscientific Digiquartz®	0.01 %

	Pressure Transducer Model 1000-500A	
Liquid Temperature	LakeShore PT-100	$\pm 0.25$ K
Heater Surface Temperature	Cryo-con S950-BB (uncalibrated)	$\pm 0.4$ K
Composition	Varian CP-3800 GC	0.6 %
Heat Flux	HP 6438B DC Power Supply	$\pm 0.25$ V, $\pm 0.025$ A

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The temperature of the liquid was measured using a temperature rake which consists of four platinum resistance thermometers (PRT) vertically spaced approximately 2.5 cm apart. Two PRTs were located below the cartridge heater and two PRTs were above the heater. The bulk sea temperature was determined by averaging the two PRT measurements that were below the heater which generally agreed within 0.1 K. The PRTs above the heater were used to measure the thermal gradient that occurred above the heater due to natural convection.

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The test cell had an internal diameter of 8.3 cm and a depth of 12.2 cm. The submarine was represented by a 5 cm long, 0.76 cm diameter cartridge heater. The heater surface temperature was measured by a silicon diode that was thermally anchored to the flat end of the heater. The power supplied to the heater was controlled using a DC power supply. Initial measurements were conducted at near steady state conditions by increasing the heater power in 2 volt increments and waiting at least 5 minutes until the liquid and heater temperature stabilized. If effervescence did not occur, heater power was increased in 2 volt increments until effervescence occurred. Later measurements were conducted by increasing the heater power rapidly; 2 volts every 30 seconds until effervescence occurred in order to minimize the effect of trace amounts of non-visible, dissolved gas coming out of solution during the progression towards effervescence. Additional information on the two measurement methods is provided in Section 3.

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Effervescence was detected optically using a video camera and borescope as shown in Figure 1. The borescope allowed the video lens to pass into the test cell and maintain a hermetic seal. As a result, the end of the borescope was subject to the condition of the fluid being measured. This led to poor resolution for a few of the measurements due to fogging. A large uncertainty in the effervescence measurements is determining when effervescence occurs; similar to the different boiling regimes, there are varying degrees of effervescence. Effervescence was determined at the point of visible bubbles.

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The composition of the liquid mixture at effervescence was obtained using a liquid trap. The liquid trap consisted of a section of brass pipe connected to the test cell through a 0.625 mm (0.25 inch) copper tube. A normally open solenoid valve was located between the test cell and the brass pipe allowing liquid to move freely between the liquid trap and the test cell. It was assumed that there was no stratification between the liquid in the test cell and the liquid trap

140 because of the turbulence caused by nitrogen bubbling. Once effervescence occurred, the  
141 solenoid valve was closed separating the liquid in the trap from the liquid in the test cell. The  
142 liquid in the liquid trap was heated until it was completely vaporized. Vaporization was ensured  
143 by heating the liquid trap above the saturation temperature of liquid ethane, which is the highest  
144 of the three components. The vapor was collected in a sampling cylinder. After the liquid was  
145 completely vaporized, the gaseous mixture was extracted and collected in a 1 liter multilayer gas  
146 sampling bag where it was analyzed via gas chromatography. For redundancy, three separate gas  
147 sampling bags were filled and analyzed for the composition measurement for each data point. A  
148 Varian CP-3800 GC system with a thermal conductivity detector was used to quantify nitrogen  
149 and a flame ionization detector was used to quantify the methane and ethane gases. The gas  
150 chromatograph utilizes a Silcosteel HaysSepQ 80/100 mesh packed column (5.5m X 3.175mm;  
151 Supelco). The method used for this analysis incorporated a 10 $\mu$ L stainless steel injection loop  
152 controlled by a Valco switching valve installed in the oven. The column oven was held isocratic  
153 at 80 °C for 8 min, and the helium carrier gas had a 65 mL/min flow rate (1.448 bar).  
154 Calibrations were made with certified standards (%v/v) of at least three levels for nitrogen (5 %  
155 up to 20 %), methane and ethane (15 % up to 100 %) having a certified accuracy of 5 %. The  
156 uncertainty associated with the certified gas standards and calibration curves were not accounted  
157 for in the reported composition uncertainties. The linear calibration curves for methane, ethane,  
158 and nitrogen had a coefficient of determination (R-squared value) greater than 0.9998 for each  
159 component suggesting the uncertainty of the certified gas standards was much less than the  
160 provided accuracy of 5 mol %. The only error considered in the reported composition  
161 uncertainties is the standard deviation from conducting composition analysis in duplicate from  
162 three gas sampling bags that were collected for each data point. The composition accuracy  
163 reported in Table 1 was founded using the largest reported standard deviation.

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165 The solenoid valve used in the liquid trap would occasionally experience leakage, which could  
166 ultimately bias the composition of the measurements. Measurements with detectable leakage  
167 have been noted. To reduce the effects of valve leakage, an effort was made to equalize the  
168 pressure on each side of the valve to reduce the flow potential.

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### 170 **3. Effervescence Measurements**

171 Measurements were conducted for liquid temperatures ranging from 92 K to 96 K and varying  
172 methane-ethane-nitrogen compositions to cover the range of sea conditions that exist on Titan.  
173 Pressures were varied from 1.5 bar to 4.5 bar to account for varying sea depths. The twelve  
174 effervescence measurements and respective uncertainties are presented in Table 2. The reported  
175 expanded uncertainties,  $U = ku_c$  where  $u_c$  is the combined standard uncertainty, were  
176 determined using a coverage factor of 2 ( $k = 2$ ). Thus the expanded uncertainties have a 95%  
177 level of confidence as recommend by the National Institute of Standards and Technology (Taylor  
178 and Kuyatt, 1994). The individual uncertainties and sources of errors for each of the  
179 experimental measurements have been discussed by Richardson (2017).

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181 Table 2: Effervescence measurements of methane-ethane-nitrogen mixtures.

Measurement	Methane [mol %]	Ethane [mol %]	Nitrogen [mol %]	Pressure [bar]	Liquid Temp. [K]	Heater Surface Temp. [K]	Heat Flux at Bubble Incipience [kW/m <sup>2</sup> ]
1 <sup>a</sup>	87.1 ±0.3	0.0	12.9 ±0.3	1.546 ±0.007	96.2 ±0.5	101.4 ±0.8	10.830 ±1.435
2	87.7 ±0.2	0.0	12.3 ±0.2	1.65 ±0.01	93.5 ±0.5	100.2 ±0.8	13.029 ±1.639
3 <sup>a</sup>	82.5 ±0.5	0.0	17.5 ±0.5	1.782 ±0.007	95.9 ±0.5	99.6 ±0.8	3.257 ±0.819
4	72.3 ±0.5	0.0	27.7 ±0.5	4.53 ±0.08	97.0 ±0.5	104.1 ±0.8	17.915 ±1.806
5 <sup>b</sup>	0.0	97.3 ±0.1	2.7 ±0.1	1.73 ±0.05	103.6 ±0.6	118.7 ±0.8	28.810 ±2.381
6	0.0	94.6 ±0.6	5.4 ±0.6	4.41 ±0.05	92.5 ±0.5	107.2 ±0.9	24.559 ±2.136
7	50.6 ±0.3	44.2 ±0.2	5.2 ±0.2	1.85 ±0.01	97.8 ±0.5	108.7 ±0.8	18.729 ±1.887
8	57.5 ±0.2	37.2 ±0.1	5.3 ±0.2	1.561 ±0.007	92.7 ±0.5	107.8 ±0.8	26.448 ±2.298
9	47.6 ±0.4	30.9 ±0.4	21.5 ±0.3	3.21 ±0.02	91.9 ±0.5	97.8 ±0.8	11.074 ±1.396
10	24.9 ±0.1	48.3 ±0.4	26.8 ±0.4	3.44 ±0.03	91.9 ±0.5	94.2 ±0.8	2.475 ±6.56
11	27.0 ±0.5	61.4 ±0.5	11.6 ±0.5	3.85 ±0.03	91.8 ±0.5	98.8 ±0.8	10.244 ±1.394
12 <sup>a</sup>	30.2 ±0.3	63.9 ±0.3	5.9 ±0.3	2.133 ±0.007	93.6 ±0.5	111.6 ±0.8	31.758 ±2465

<sup>a</sup> Leakage through the solenoid valve may have biased the composition more than the reported uncertainty.

<sup>b</sup> Unable to achieve effervescence.

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184 Measurements 1-5 and 7 allowed the temperature of the liquid mixture and heater surface to  
185 stabilize before increasing the heater power as discussed in Section 2. As a result, several of the  
186 liquid temperatures were higher than anticipated because the cryocooler was unable to remove  
187 the amount of heat that was being added to the liquid by the cartridge heater while maintaining  
188 the desired temperature. Measurements 6 and 8-12 rapidly increased the heater power until  
189 effervescence occurred.

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191 Measurements 3 and 10 show a significantly lower heat flux and temperature differential  
192 between the heater surface and liquid. Effervescence for measurement 3 was recorded for a very  
193 small stream of bubbles coming from a single point on the heater. Measurement 10 was  
194 conducted under poor visibility and was determined when there was significant disturbance to  
195 the liquid-vapor interface. This could have led to this point being conducted prematurely. Videos  
196 of effervescence for each of the data points are publically available to visualize the variability in  
197 effervescence for each measurement at <http://hdl.handle.net/2376/12183>. Still images from the  
198 video showing effervescence for measurement 11 and 12 are shown in Figure 2.  
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202 Figure 2. Images of effervescence during measurement 11 (left) and measurement 12 (right).  
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204 Measurements 1, 3, and 12 experienced detectable leakage through the solenoid valve. This  
205 could have potentially biased the composition measurement as liquid from the test cell could  
206 flow into the liquid trap and vice versa. As a result the uncertainty associated with the  
207 composition of these measurements may be higher than the reported values.  
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209 Effervescence was not achieved for measurement 5. The upper voltage limit of the power supply  
210 was reached before effervescence occurred. However insight was still gained by measurement 5  
211 which showed severe convection currents near the cartridge heater which may have a negative  
212 impact on submarine operations and instrument readings. Nonetheless, bubble incipience data in  
213 Table 2 are consistent with solubility limits for liquid ethane and liquid methane; due to the  
214 lower solubility of nitrogen in ethane, there are fewer bubbles available to come out of solution  
215 requiring more heat for bubble incipience. The converse is true for higher liquid methane seas.

216 Furthermore, waste heat flux at bubble incipience is higher at higher pressures and colder liquid  
217 temperatures.

218  
219 From the Phase 1 Titan Submarine (Hartwig et al., 2016), the waste heat flux into the Titan seas  
220 was estimated to be  $370 \text{ W/m}^2$  from a detailed thermal balance between radioisotope generator  
221 power source, internal insulation, and coolant distribution network. Examination of Table 2  
222 shows that the lowest recorded heat flux at the point of bubble incipience is nearly an order of  
223 magnitude higher. This implies that the current submarine design has nearly an order of 10 safety  
224 factor on the resultant waste heat needed to produce nitrogen bubbles which would interfere with  
225 science instruments or propellers.

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#### 227 **4. Freezing Point Depression Measurements**

228 Upon completion of the effervescence measurements, two freezing point depression  
229 measurements were conducted. Though ice buoyancy and formation relevant to Titan has  
230 previously been investigated (e.g. Thompson, 1985; Roe and Grundy, 2012; Prokhvatilov and  
231 Yantsevich, 1983; Hofgartner and Lunine, 2013), these measurements were necessary to verify  
232 predictive models in the literature and to provide experimental data for current Titan sea property  
233 models. The same experimental setup was used for the freezing measurements. The experimental  
234 procedure was also kept the same except instead of adding heat to achieve effervescence, the test  
235 cell and liquid were allowed to continue to cool until ice began to form. The results of the  
236 freezing point measurements are presented in Table 3.

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Table 3. Freezing point depression measurements.

<b>Measurement</b>	<b>Methane [mol %]</b>	<b>Ethane [mol %]</b>	<b>Nitrogen [mol %]</b>	<b>Pressure [bar]</b>	<b>Liquid Temp. [K]</b>
F1	$61.0 \pm 0.1$	$25.6 \pm 0.1$	$13.4 \pm 0.1$	$0.290 \pm 0.007$	$71.5 \pm 0.5$
F2	$46.9 \pm 0.2$	$45.3 \pm 0.1$	$7.9 \pm 0.3$	$0.517 \pm 0.007$	$74.0 \pm 0.5$

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240 The freezing point depression measurements show dramatic subcooling below the triple point  
241 temperatures of methane (90.7 K) and ethane (90.4 K) (Lemmon et al., 2013). Freezing point F2  
242 was verified visually using the video camera and borescope. A still image of freezing during  
243 measurement F2 is shown in Figure 3. The black circle in Figure 3 shows a large white mass of  
244 ice in the upper left section of the test cell. The full video is available at  
245 <http://hdl.handle.net/2376/12183> and shows the growth of a solid white ice ball within the test  
246 cell at a temperature of 74 K and pressure of 0.517 bar.

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Figure 3. Image of freezing occurring during measurement F2.

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251 The first freezing point measurement F1 could not be confirmed visually due to severe fogging  
252 on the borescope lens. Instead freezing was determined using the temperature and pressure  
253 measurements. The onset of freezing was determined when the pressure stopped decreasing  
254 similar to what was observed by Guildner et al. (1976). At the onset of freezing the temperature  
255 of the liquid stopped decreasing and stayed constant as the heat extracted by the cryocooler was  
256 absorbed by the latent heat of fusion which is responsible for solidification. The pressure and  
257 temperature showed similar behavior to freezing point F2.

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259 The freezing liquid line was depressed more than 15 K below the triple point temperatures of  
260 pure ethane. Though there are only two measurements, this data suggests that freezing will occur  
261 at higher temperatures for ethane-rich mixtures. This observation is consistent with historical  
262 observations.

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## 264 5. Conclusions

265 The likelihood of effervescence in a methane-ethane-nitrogen mixture increases with increased  
266 nitrogen content. Effervescence in ethane-nitrogen mixtures was only achieved for temperature  
267 differences between the submarine surface and liquid greater than 14 K. It was discovered that  
268 nitrogen will slowly come out of solution without causing effervescence if heating is done slowly  
269 over several minutes. When the heater power was ramped slowly, the pressure in the sealed test  
270 cell would continue to rise as the temperature of the liquid increased due to nitrogen coming out  
271 of the liquid. Furthermore, the temperature rake measured a significant thermal gradient between  
272 liquid below the heater and liquid above the heater. This suggests that heat and bubbles radiating  
273 out from the submarine will rise up and away from the submarine. These effects occur at lower  
274 heat fluxes and smaller temperature differences between the sea and submarine surface for  
275 methane-rich mixtures. Nevertheless, results show that there is an appreciable safety factor on  
276 the resultant heat flux into the liquid before the point of bubble incipience for current submarine  
277 waste heat fluxes.

278

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