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

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

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

2. Experimental Design

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

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

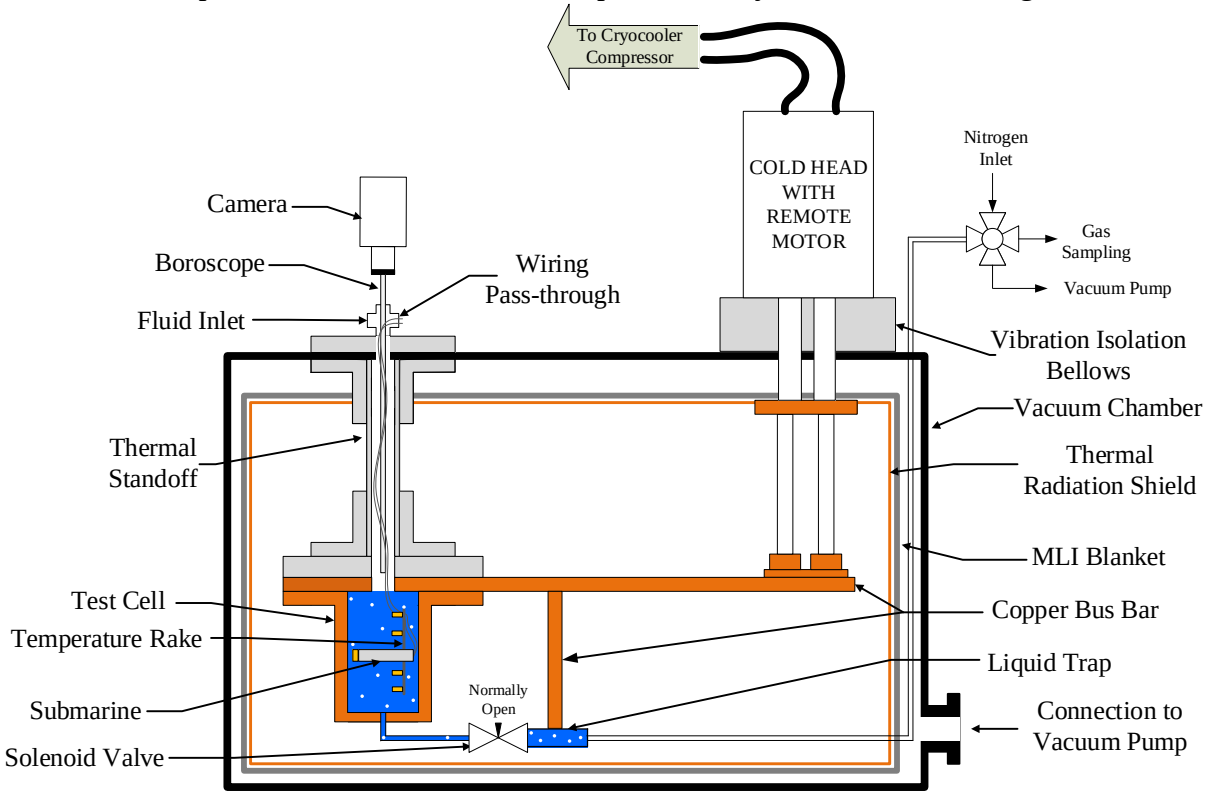

Figure 1. Experimental system used to conduct effervescence measurements.

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

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	±0.25 K
Heater Surface Temperature	Cryo-con S950-BB (uncalibrated)	±0.4 K
Composition	Varian CP-3800 GC	0.6 %
Heat Flux	HP 6438B DC Power Supply	±0.25 V, ±0.025 A

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.

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.

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.

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

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

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

3. Effervescence Measurements

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

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^2]**
1[a]	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[a]	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[b]	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[a]	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

[a] Leakage through the solenoid valve may have biased the composition more than the reported uncertainty.

[b] Unable to achieve effervescence.

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

Measurements 3 and 10 show a significantly lower heat flux and temperature differential between the heater surface and liquid. Effervescence for measurement 3 was recorded for a very small stream of bubbles coming from a single point on the heater. Measurement 10 was conducted under poor visibility and was determined when there was significant disturbance to the liquid-vapor interface. This could have led to this point being conducted prematurely. Videos of effervescence for each of the data points are publically available to visualize the variability in effervescence for each measurement at http://hdl.handle.net/2376/12183. Still images from the video showing effervescence for measurement 11 and 12 are shown in Figure 2.

Figure 2. Images of effervescence during measurement 11 (left) and measurement 12 (right).

Measurements 1, 3, and 12 experienced detectable leakage through the solenoid valve. This could have potentially biased the composition measurement as liquid from the test cell could flow into the liquid trap and vice versa. As a result the uncertainty associated with the composition of these measurements may be higher than the reported values.

Effervescence was not achieved for measurement 5. The upper voltage limit of the power supply was reached before effervescence occurred. However insight was still gained by measurement 5 which showed severe convection currents near the cartridge heater which may have a negative impact on submarine operations and instrument readings. Nonetheless, bubble incipience data in Table 2 are consistent with solubility limits for liquid ethane and liquid methane; due to the lower solubility of nitrogen in ethane, there are fewer bubbles available to come out of solution requiring more heat for bubble incipience. The converse is true for higher liquid methane seas.

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

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

4. Freezing Point Depression Measurements

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

Table 3. Freezing point depression measurements.

Measurement	Methane [mol %]	Ethane [mol %]	Nitrogen [mol %]	Pressure [bar]	Liquid Temp. [K]
F1	61.0 ±0.1	25.6 ±0.1	13.4 ±0.1	0.290 ±0.007	71.5 ±0.5
F2	46.9 ±0.2	45.3 ±0.1	7.9 ±0.3	0.517 ±0.007	74.0 ±0.5

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

Figure 3. Image of freezing occurring during measurement F2.

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

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

5. Conclusions

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

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