

Measurements of the Vapor Pressure of Liquid Hydrazine above Its Normal Boiling Point

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Abstract

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1. Introduction

Hydrazine (N_2H_4) is a propellant that is used in propulsion systems for spacecraft due to its ability to be utilized for both monopropellant applications, with the aid of a catalyst, and as a hypergolic bipropellant, when combined with the oxidizer nitrogen tetroxide. Since it is the propellant of choice for in-space thruster applications, a detailed knowledge of the thermal properties of liquid hydrazine is of major interest to aid in the designing of propulsion systems. The numerical analysis of propulsion system designs typically requires the use of a mathematics-based equation of state for hydrazine. There are several equations of state previously published in the literature for hydrazine [1]–[3]. To validate the performance of an equation of state for a substance a detailed knowledge of the vapor pressure is typically required.

The vapor pressure of hydrazine has been experimentally measured many times previously [4]–[11]. A good review of the current state of the experimental vapor pressure data published in open literature was conducted by Giordano [12]. The literature has several analytical forms of the vapor pressure correlation function for hydrazine as well. Giordano [12] noted that the vapor pressure interpolating function of Das and Kuloor [13] [Eq. (1)] did an adequate job of reproducing the experimental data from 273.15 K to 653.15 K, where the temperature (T) is in K.

$$P(atm) = e^{58.7582 - \frac{0.707 \times 10^4}{T} - 7.088 \times \ln(T) + 0.00457 \times T} \quad (1)$$

Barragan et al. [2] utilized the vapor pressure interpolating function of Yaws [14] [Eq. (2)] in developing their equation of state.

$$P(Torr) = 10^{60.878 - \frac{3880.3}{T} - 20.575 \log(T) + 0.015585 \times T - 5.0525 \times 10^{-6} \times T^2} \quad (2)$$

Schmidt has a summary that includes several other hydrazine correlation functions than the two shown above. These other correlation functions have a more limited use within the community [15]. Both Giordano [12] and Schmidt [15] noted that the data set from reference West and Hull [6] doesn't appear in the cited material of de Bruyn even though it was credited to that source. Giordano thus concluded that while the data set is fit well by the interpolating functions, the exact origin is uncertain and it is not known if the data is really experimental in origin or simply an extrapolation of existing experimental data. This is unfortunate as the data set in reference 6 includes nearly all of the experimental data recorded for hydrazine's vapor pressure above the normal boiling point (387.3 K) [7][16]. Tests of the only other existing experimental data points above the normal boiling point were conducted back in the 19th century by de Bruyn. Collecting experimental data above the normal boiling point is of interest for calculating the liquid vapor interaction of hydrazine in high-pressure environments such as those found in the injector and film-cooling environment of an in-space propulsion system. Schmidt [15] notes that the data above the normal boiling point has likely not been measured again due to the unstable nature of hydrazine, which makes it an explosion hazard. In addition, hydrazine vapor tends to decompose at these elevated temperatures on most surfaces, making such data collection challenging.

The current experiments measured the vapor pressure above the normal boiling point for the first time since 1896. The temperature range covered was 324 K, which is below the normal boiling point, to 414 K. Decomposition was observed to occur in the vapor phase at elevated temperatures, which complicated the data acquisition and interpretation at these elevated temperatures. The current data set agreed well with the previous experimental measurements and the adjusted values for hydrazine's vapor pressure suggested by Mitchell et al. [16]. Mitchell et al. previously suggested a small shift to the vapor pressure curve at elevated temperatures based on their Peng-Robinson equation of state analysis. A new vapor pressure

correlation function was produced for hydrazine that fits well with both the current experimental data, the previously reported experimental data, and the adjusted values of Mitchell et al.

2. Experimental

A schematic of test apparatus used in the vapor pressure experiments is shown in Figure 1. The test apparatus was composed of a vapor pressure chamber made of titanium, tubing to a nitrogen supply, and a vacuum system. Titanium was chosen for the construction of the vapor pressure chamber due to the high exothermic threshold temperature (479 K) of hydrazine with titanium surfaces [15]. Although the temperature of the experiment should remain below the autoignition temperature, the internal volume (26 ml) of the vapor pressure chamber was designed to minimize the amount of liquid hydrazine needed just in case an autoignition event occurred at elevated temperature. The vapor pressure chamber was designed to handle pressures of 10000 PSI or greater in case an autoignition event did occur. The temperature for the experiment was regulated by a temperature controller that controlled the current sent to four cartridge heaters placed inside a copper block. The vapor pressure chamber was press fit into the copper heating block. Heating tape was used on the top part of the vapor pressure chamber to reduce thermal gradients. The temperature was monitored by a K-type thermocouple attached to the wall of the vapor pressure chamber. A piezo-resistive pressure transducer directly sampled the pressure inside the vapor pressure chamber.

An experiment was conducted by placing the test liquid (0.6 ml) inside the vapor pressure chamber. The vapor pressure chamber was then evacuated. In general, three freeze pump thaw cycles were used to remove air and any dissolved gases that may have been in the hydrazine. The temperature controller was then set to the desired test temperature. The vapor pressure chamber was allowed to reach a temperature equilibrium, and then the pressure was recorded. The temperature was then changed, and equilibrium reestablished before the next data point. There were three major concerns with the current test apparatus. The first was the calibration of the pressure gauge over the temperature range of the experiment, and the second was ensuring that the measure of the temperature at the apparatus wall properly corresponded to the temperature of the liquid. The third concern was hydrazine decomposition in the vapor phase.

In order to understand the effects of the first concern—that the change in the calibration of the pressure transducer with changes in temperature was well characterized—the pressure sensor was tested at several elevated temperatures. While the pressure sensor was chosen because of its stated capability to work at both high pressures (up to 200 psi) and high temperatures (up to 533 K), the drift in the calibration was uncertain since the pressure sensor was calibrated at room temperature. A reference gauge (MKS) was set up such that it would remain at room temperature while the vapor pressure chamber's temperature was changed. Figure 2 shows that the calibration doesn't change significantly over the range of the temperatures measured in the experiment compared to the room temperature reference gauge. While this is a positive result, the overall point-to-point variance in the pressures measured by the high-temperature gauge compared to the MKS gauge is a little disconcerting. The standard deviation of the difference in the pressure between the two gauges was measured to be 0.35 PSI. This is higher than would be ideal. However, the average error in Figure 2 is only 0.0016 PSI. Demonstrating that if a large enough data set of measurements is taken even with the current gauge, then the average scatter in vapor pressure curve data will be sufficiently averaged out.

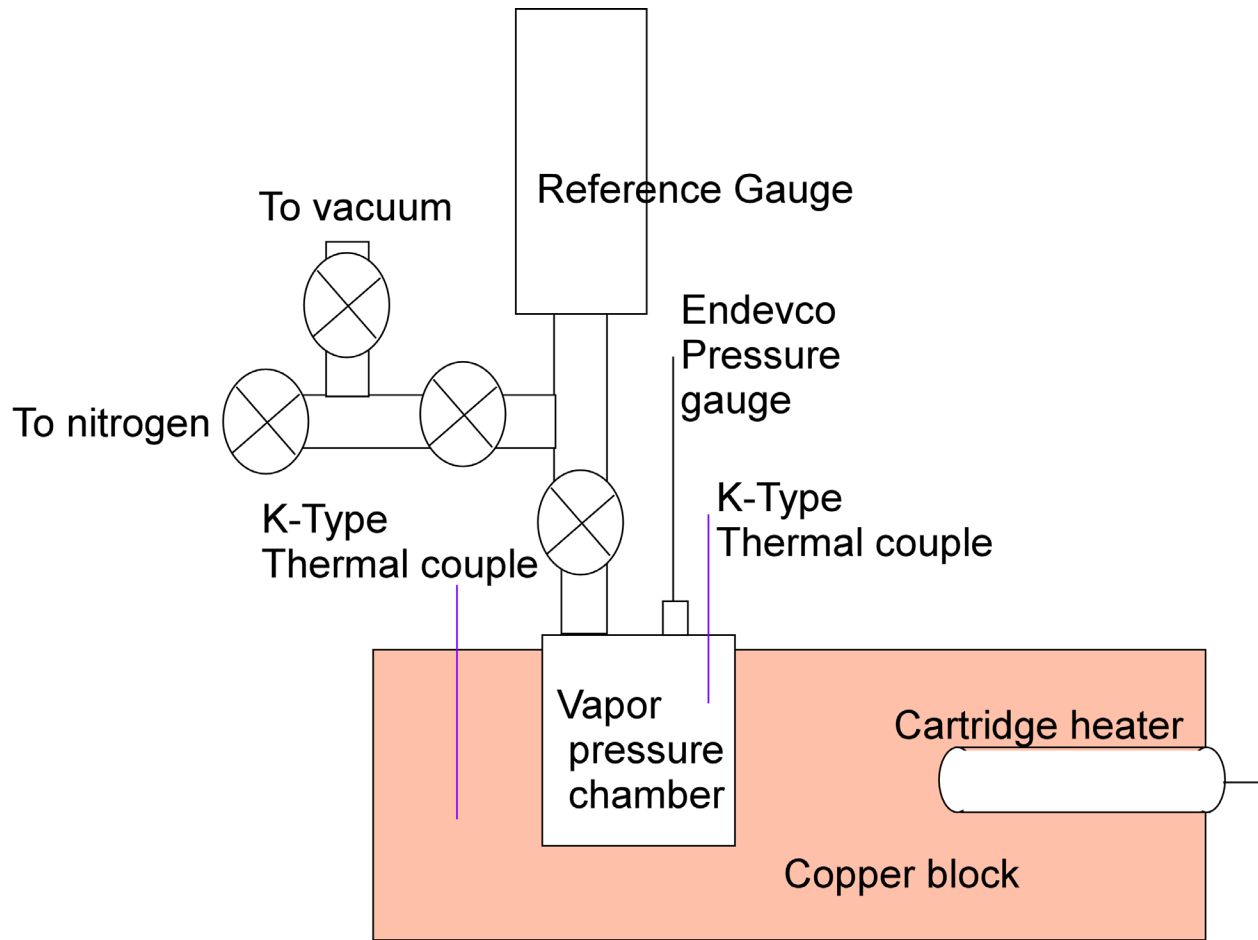


Figure 1. The setup for the vapor pressure measurement test.
The vapor pressure chamber is placed into a copper heater block.

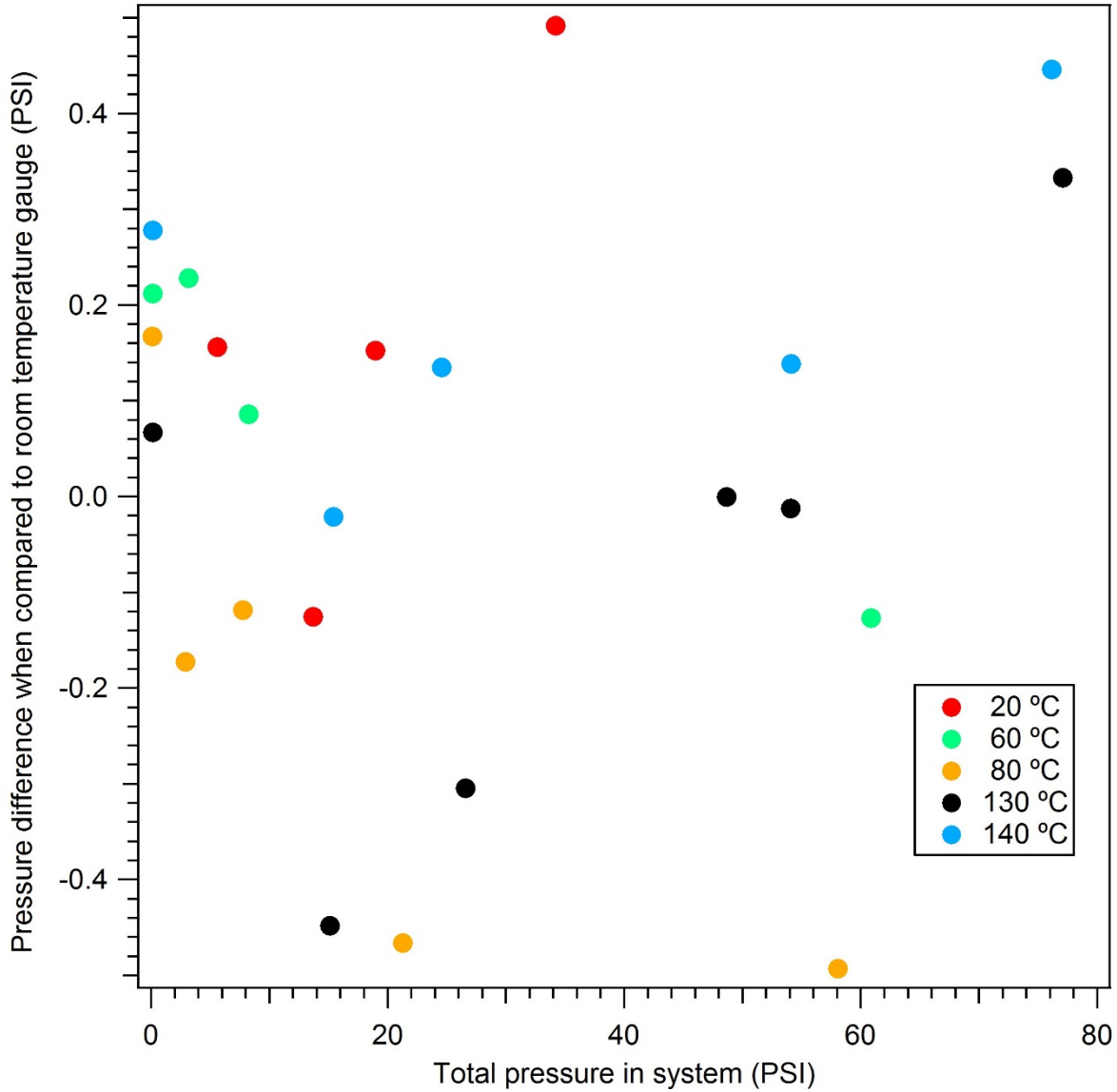


Figure 2. The difference in the pressure inside the vapor pressure chamber measured by the high-temperature gauge versus a low-temperature gauge outside the vapor pressure chamber at different total pressures. Overall there is little noticeable temperature effect on the high-temperature pressure gauge.

The second concern—if the recorded temperature during the thermal equilibrium was that of the liquid inside the chamber or if there was a slight thermal gradient between the chamber wall and the liquid temperature—was handled by using water as a calibration liquid. The literature vapor pressure of water [17] was used to calibrate the temperature. The temperature recorded by the thermocouple was compared to the predicted temperature based on the pressure measured inside the chamber when water was used. Figure 3 shows the current water data compared to the literature data. A small growing offset in the current data was observed compared to the literature data as the temperature was increased. Both the literature water data and the current measured data from 319.15 K to 423.15 K were fit by an Antoine equation [Eq. (3)].

$$T_K = \left(\frac{-B}{\text{Log}(P_{\text{PSI}}) - A} \right) - C \quad (3)$$

The two resulting fits on Figure 3 were

$$T(K) = \left(\frac{-1275.66}{P-5.66976} \right) - (-87.2443) \quad (4)$$

$$T(K) = \left(\frac{-1573.6}{P-6.1045} \right) - (-54.342) \quad (5)$$

for the current data [Eq. (4)] and the literature data [Eq. (5)].

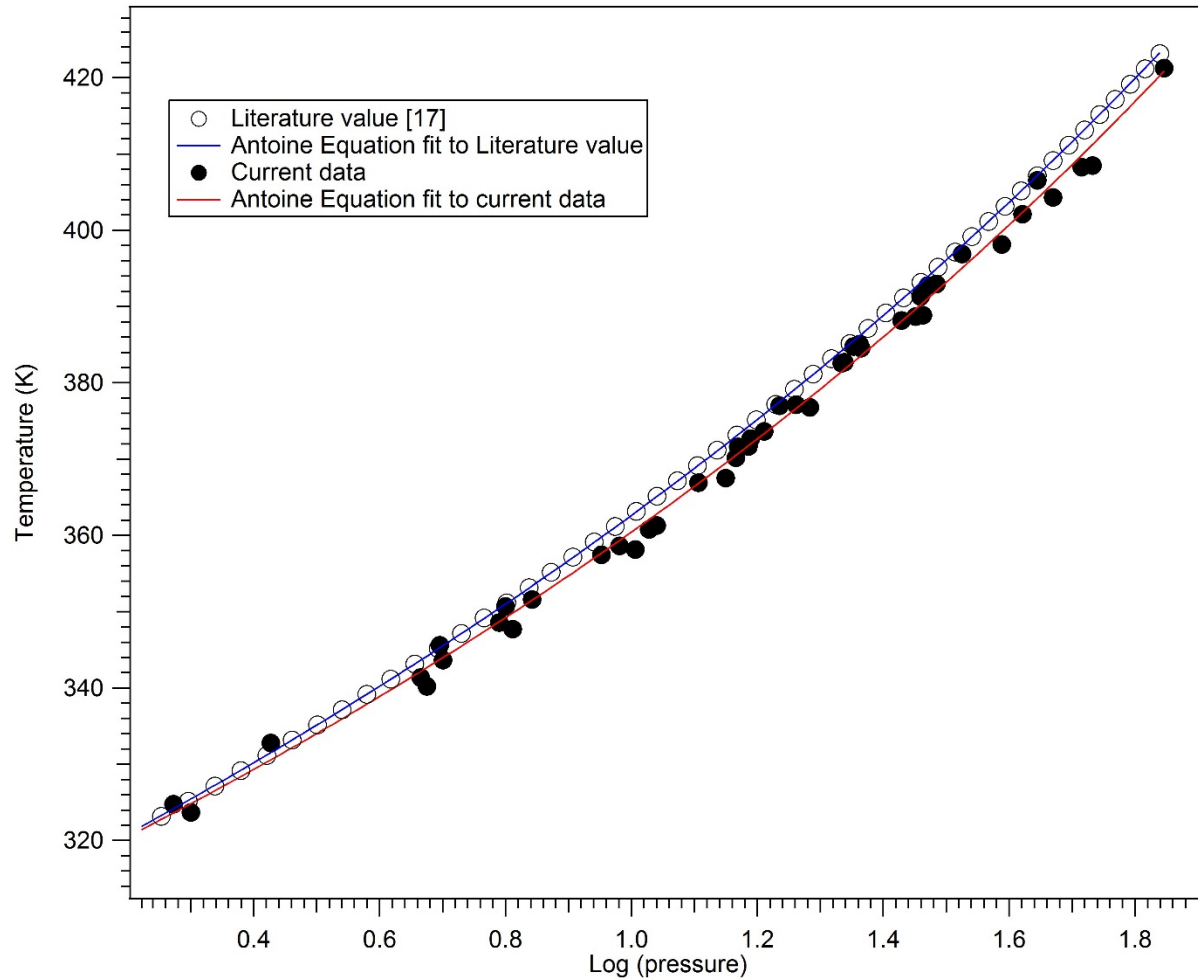


Figure 3. The temperature as a function of the LOG[pressure (PSI)]. The open circles are the literature values, and the closed circles are the data taken from the thermocouple and pressure sensor on the vapor pressure apparatus. The blue curve is an Antoine equation fit to the literature water data. The red curve is an Antoine equation fit to the water data.

A temperature correction function was produced by fitting the difference in the water literature temperature—predicted by Eq. (5)—versus the temperature predicted by the Antoine equation fit to the current data in Figure 3 [Eq. (4)]. Figure 4 shows that the temperature difference increased with increased temperature as expected if a slight temperature gradient existed in the system. The correct temperature of the liquid can be obtained by adjusting the temperature on the thermocouple by the following equation:

$$T_{\text{adjusted}} = T_{\text{thermal couple}} + T_{\text{correction}} \text{ (K)} = T_{\text{thermal couple}} - 47.10 \pm 0.07 + 0.2407 \pm 0.0004 * T(\text{K}) - 0.00028883 * T^2(\text{K}^2) \quad (6)$$

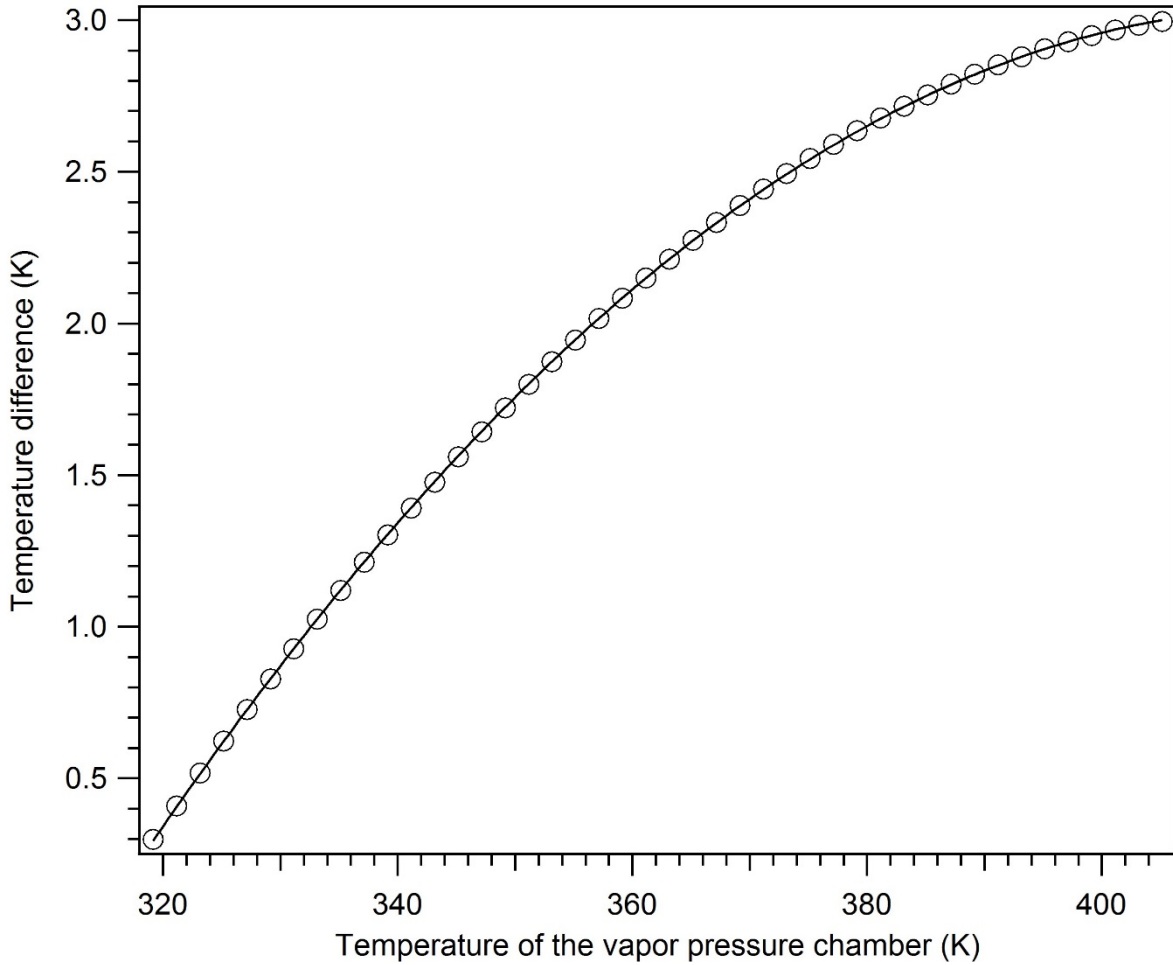


Figure 4. The difference in temperature between the curve predicted by the data [Eq. (5)] and the literature temperature for a given vapor pressure versus the temperature measured by the thermal couple on the vapor pressure apparatus. The black line is a curve fit [Eq. (6)] to the data.

Figure 5 has the original water data plotted versus literature data and the water data adjusted by Eq. (6) to correct for the temperature gradient in the system. The water data adjusted by Eq. (3) was fit to the following equation:

$$P(\text{PSI}) = 14.695 * e^{(29.882 - \frac{5743}{T} - 2.5184 * \text{LN}(T) - 0.0011253 * T)} \quad (7)$$

The equation fits the current water data with a standard deviation of 0.13 PSI from 320 K to 423 K.

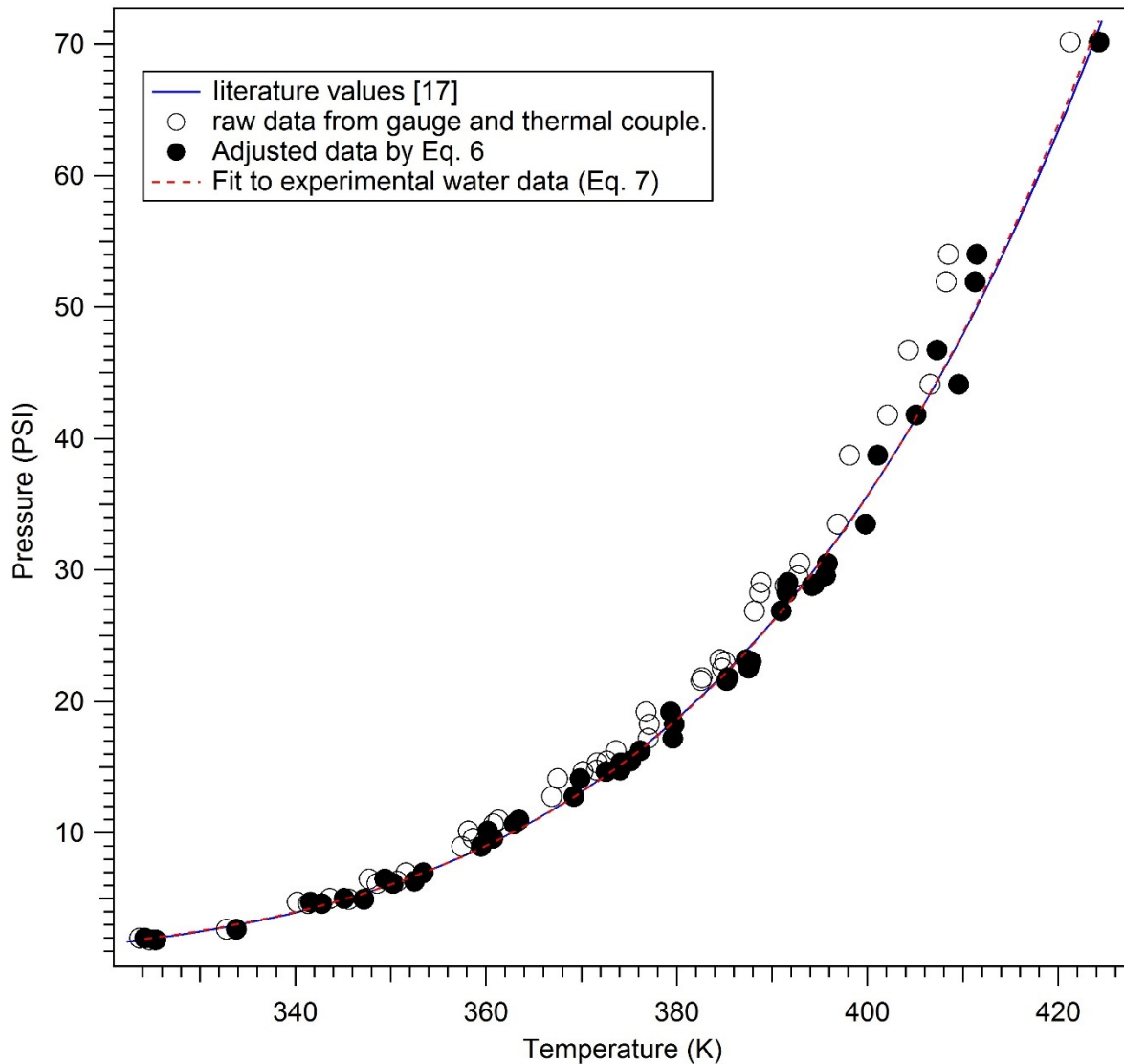


Figure 5. The vapor pressure of water as a function of temperature. The open circles are the original data, and closed circles are the adjusted data to account for the temperature gradient in the vapor pressure apparatus temperature measuring point and the liquid water. The blue curve is the literature data for water vapor [17], and the red curve is a fit to the present adjusted data [Eq. (7)].

In addition to the complications that arose from calibration of the apparatus, there is one additional concern when hydrazine is used in the apparatus. The hydrazine vapor was observed to decompose on the time scale required to reach temperature equilibrium. This required the valve to the vacuum pump to be cycled after reaching temperature equilibrium. Cycling the valve allowed the vapor above the liquid to be refreshed prior to each pressure reading. This is necessary to acquire accurate vapor pressure measurements since the decomposition produces gaseous products such as nitrogen and ammonia. This procedure was also repeated for several water sample tests to ensure that evacuating the chamber prior to a reading didn't add any unforeseen complications to the results. The water data was not noticeably different when data was collected in this fashion. The major source of noise observed in the test apparatus comes from the uncertainty in the pressure gauge itself used in the experiments.

The water used in the current experiments was deionized 18 M Ω cm obtained from an in-house laboratory source. The hydrazine used in the current experiments was purchased from Sigma-Aldrich Co. (98+% purity). The nitrogen (Airgas) purity was 99.998%.

3. Results and Discussion

The results for the hydrazine experiments are shown in Figure 6 and Table 1. The current data agrees with the two older hydrazine correlation functions with an average absolute error of 0.61 PSI from that prediction by using Eq. (1) and 0.66 PSI by using Eq. (2). A fit of the current data by using an equation similar to Eq. (1)–Eq. (8) with the addition of the critical point—falls extremely close to the fit of [13], as seen in Figure 6.

$$P(\text{PSI}) = e^{59.5786 - \frac{7015.15}{T} - 6.77418 \times \ln(T) + 0.00418762 \times T} \quad (8)$$

The average error for the three correlation functions—Eqs. (1), (2), and (8)—to the previous literature data is shown in Table 2. This current fit is slightly better at fitting all the previous literature data (excluding [9] from these fits since it was poorly fit by all functions and reference 6, which has an uncertain origin) than Eq. (1) [13] but has a slightly greater average error than Eq. (2) [14]. When the current data is added to the dataset, the average error for the three correlation functions is very similar. Hence, the current data set isn't accurate enough to distinguish between the three fitting functions for increased accuracy at higher temperatures.

Mitchell et al. [16] found that the Peng-Robinson equation of state was the most accurate method for determining the liquid-vapor fugacity coefficients for hydrazine. They noted that fugacity coefficients follow a 45-degree line for only a short segment of their evaluation before deviating. To improve their calculations, they made an adjustment of the vapor pressure curve used to produce fugacity coefficients that more closely follow the 45-degree line required. Using the vapor pressure predicted by the Barragan et al. vapor pressure correlation [2][14] as a starting point, they adjusted the vapor pressure in an iterative process. They adjusted the vapor pressure until the liquid and vapor phase fugacity values were equivalent, and thus a thermodynamically consistent value of the low-temperature vapor pressure could be obtained. The major change in the adjusted vapor pressure (about a three percent difference) occurs in the high-temperature region where only [6] had available data. Figure 7 shows the current hydrazine data along with Mitchell's corrected values. Qualitatively Mitchell's adjusted points do not appear much different than the values found in reference 6. The two most common vapor pressure correlation fits—Eqs. (1) and (2)—fit the data from [6] about equally well—2.82 PSI average error for Eq. (1) and 2.91 PSI for Eq. (2)—with most of the error due to poor fitting of the 623.15 K data point. Without the last data point, the average error changes to 1.41 PSI for Eq. (1) and 1.44 PSI for Eq. (2). They both do a poor job (as expected) in fitting Mitchell's vapor pressure adjustment [average error of 10.02 PSI for Eq. (1) and average error of 10.86 PSI for Eq. (2)]. However, it's not clear that fitting the data set from [6] over Mitchell's adjusted vapor pressure prediction is a valid concern, given the questionable origin of the data in [6].

The adjusted predicted vapor pressure points from Mitchell et al.'s Peng-Robinson equation of state were combined with the current data set and previous data points below the boiling point to produce a new fit that is consistent with the Peng-Robinson equation of state evaluation from Mitchell et al. [16].

$$P(\text{PSI}) = e^{98.9831 - \frac{8384.67}{T} - 13.2769 \times \ln(T) + 0.0116005 \times T} \quad (9)$$

The average error and standard deviation for the fit is shown in Table 3. Overall equation 9 does no worse fitting the old data or the current data than Eq. (1) or (2) did. The main difference is relying on the adjustment of Mitchell et al. over the data in [6] at the higher-temperature region. A five-parameter fit similar to Eq. (2) was also tried, but it didn't significantly reduce the average error.

4. Conclusion

The current set of experiments investigated the vapor pressure of liquid hydrazine above the boiling point for the first time since the 1890s. The current data set has more experimental error associated with it than would have been ideal. The current set of data also doesn't extend to high enough temperatures and pressures to hit the areas where the Peng-Robinson equation of state adjustments of Mitchell et al. showed the biggest adjustments. Thus, the difference between [16] and [6] could not be interrogated. Overall the current data set is consistent with previous prediction of the vapor pressure in the temperature region covered. The new vapor pressure correlation fit is consistent with older published data, current data, and the equation of state work by Mitchell et al.

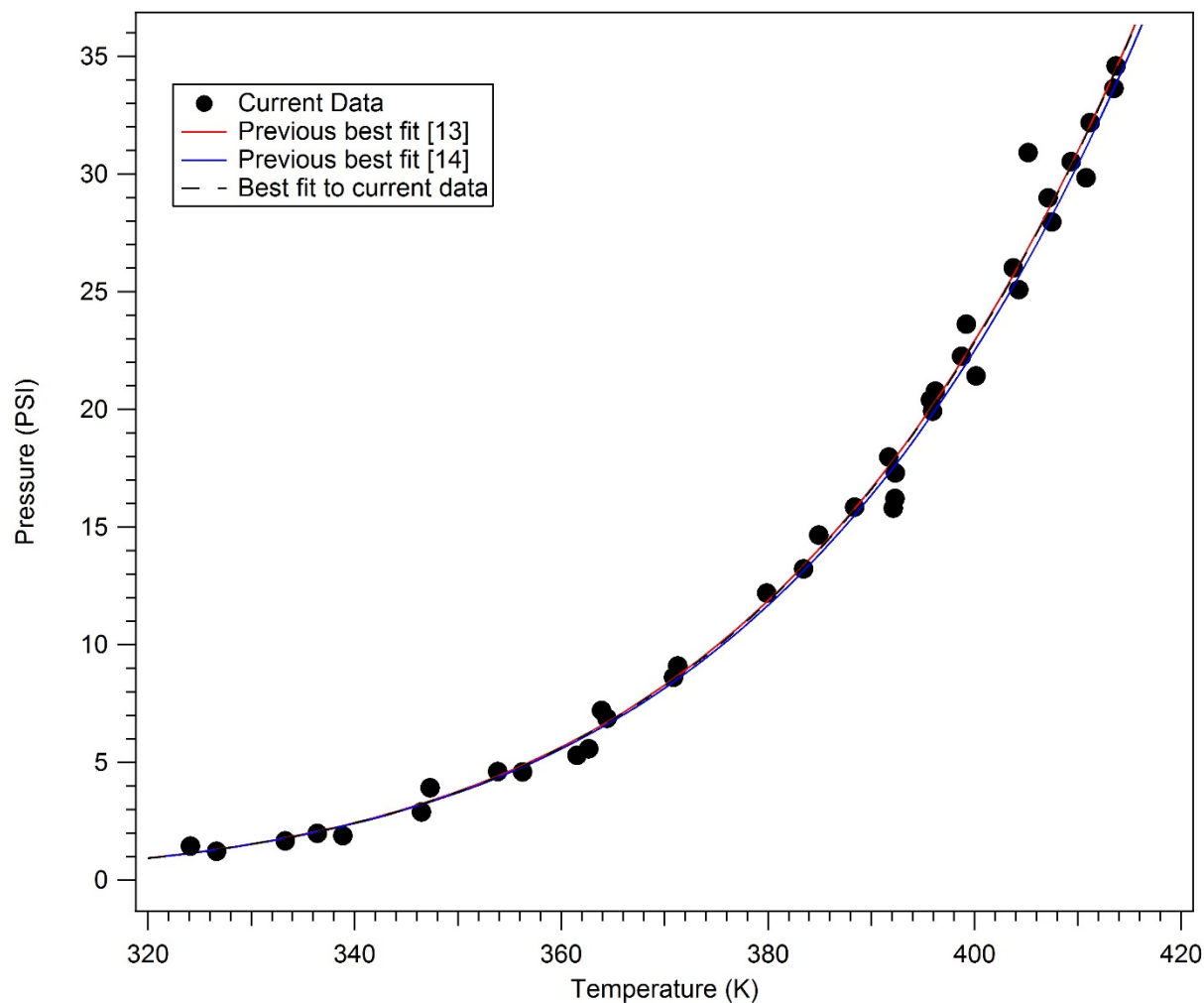


Figure 6. The measured vapor pressure of hydrazine as a function of temperature. Also shown are the previous literature fits to hydrazine data; the red curve is from [13] and the blue curve is from [14]. The dashed fit is the best fit to the current data plus the critical point.

Table 1. The Measured Liquid N₂H₄ Vapor Pressure at Various Temperatures

Temperature (K)	Vapor Pressure (PSI)	Vapor Pressure (Torr)
413.70	34.59	1788.88
413.50	33.63	1739.38
411.18	32.19	1664.63
410.78	29.84	1543.14
409.36	30.53	1578.88
407.49	27.95	1445.47
407.12	28.98	1498.66
405.19	30.91	1598.65
404.28	25.09	1297.55
403.76	26.00	1344.82
400.14	21.41	1107.20
399.20	23.62	1221.46
398.75	22.25	1150.57
396.19	20.77	1074.16
395.94	19.93	1030.58
395.74	20.41	1055.60
392.32	17.30	894.75
392.28	16.20	837.98
392.12	15.80	817.32
391.69	17.96	929.03
388.39	15.85	819.69
384.91	14.65	757.78
383.44	13.22	683.66
379.89	12.19	630.51
371.26	9.10	470.77
370.86	8.61	445.23
364.43	6.86	354.91
363.90	7.21	372.82
362.65	5.58	288.37
361.51	5.29	273.65
356.26	4.59	237.53
353.85	4.62	238.74
347.31	3.93	202.98
346.46	2.90	149.84
338.85	1.89	97.62
336.37	1.99	102.69
333.29	1.66	86.09
326.66	1.22	62.83
324.11	1.44	74.55

Table 2. The Average Error and Standard Deviation of the Previous and Current Fits to the Experimental Data. The Data from [9] Was Excluded from the Fits As Well As the Poorly Sourced [6].

Data Fit	Average Error Eq. (1) (PSI)	Standard Deviation Eq. (1) (PSI)	Average Error Eq. (2) (PSI)	Standard Deviation Eq. (2) (PSI)	Average Error Eq. (8) (PSI)	Standard Deviation Eq. (8) (PSI)
Previous literature data [4][5][7][8][10][11]	0.10	0.32	0.04	0.07	0.05	0.08
Previous literature data plus current data	0.30	0.59	0.29	0.57	0.27	0.54

Table 3. The Average Error and Standard Deviation of the Previous and Current Fit Using Mitchell's Adjustments Based on the Equation of State

Data Fitted	Average Error Eq. (1) (PSI)	Standard Deviation Eq. (1) (PSI)	Average Error Eq. (2) (PSI)	Standard Deviation Eq. (2) (PSI)	Average Error Eq. (9) (PSI)	Standard Deviation Eq. (9) (PSI)
Previous literature data [4][5][7][8][10][11]	0.10	0.32	0.04	0.07	0.04	0.07
Current data plus previous literature data	0.30	0.59	0.29	0.57	0.29	0.57
Mitchell adjustment [16]	10.02	9.72	10.86	10.00	0.27	0.54
Reference [6]	2.82	3.49	2.91	6.07	9.49	10.59

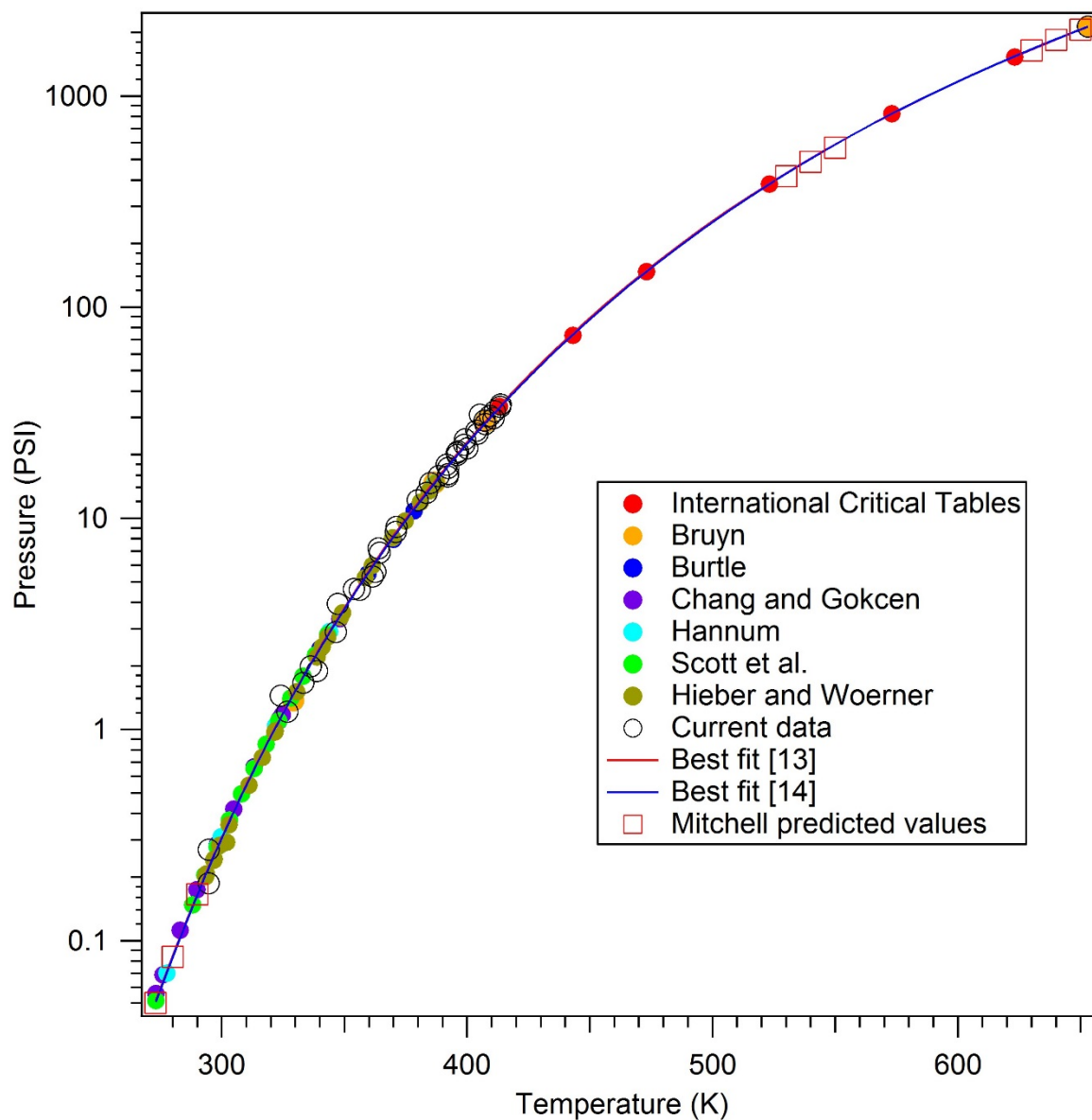


Figure 7. The log of the measured vapor pressure versus the temperature of the liquid. The corresponding experimental data and fits for each point and curve are shown in the legend.

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Technical Reports Addendum Asset Summary #2018050214275626355

Report Name: hydrazine vapor pressure

JO: 892500

First Aerospace Author / PI: Desain, John D

Created By: John D Desain

NON Aerospace MTE: *No assets reported.*

ACK322 MKS INSTRUMENTS INC. 870B12PCB2GA1

Usage Dates: 01/02/2018 - 04/01/2018

Calibration Date	Calibration Due Date	Certificate Number	Certificate Notes
04/28/2017	11/25/2018	d8e8ca85693c8748bf576c530039f73f	TMT-NORMAL

ACR123 ENDEVCO 8530C-50

Usage Dates: 01/02/2018 - 04/01/2018

Calibration Date	Calibration Due Date	Certificate Number	Certificate Notes
02/11/2016	02/11/2018	9d545f2edf7b0a4ba37fe03b38bd40ee	TMT-NORMAL
03/28/2018	08/23/2020	d3c7d122d791de419a5b3f4ebf6b936f	TMT-NORMAL

ADG311 MKS INSTRUMENTS INC. 740C52PFF2GA

Usage Dates: 01/02/2018 - 04/01/2018

Calibration Date	Calibration Due Date	Certificate Number	Certificate Notes
01/06/2017	08/05/2018	7e95d8856828834193baaae381ac7088	TMT-NORMAL

Technical Reports Addendum Asset Summary #2018050214275626355

ADJ537 NATIONAL INSTRUMENTS 9211

Usage Dates: 01/02/2018 - 04/01/2018

Calibration Date	Calibration Due Date	Certificate Number	Certificate Notes
09/08/2017	04/07/2019	a260ea34c515834c8430c1ca00f926a9	TMT-NORMAL

AED933 MEGGITT ENDEVCO 8523-200

Usage Dates: 01/02/2018 - 04/01/2018

Calibration Date	Calibration Due Date	Certificate Number	Certificate Notes
12/14/2017	01/13/2019	7abce92339586e488f71aafc6cfbbe7f	TMT-NORMAL

AED934 MEGGITT ENDEVCO 8530C-50

Usage Dates: 01/02/2018 - 04/01/2018

Calibration Date	Calibration Due Date	Certificate Number	Certificate Notes
12/22/2017	01/20/2019	9df416c2ba29344aa004381a4c698639	TMT-NORMAL

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