

Vapor Pressure and Enthalpy of Vaporization of 2-Amino-3-methylpyridine

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The vapor pressure of 2-amino-3-methylpyridine was measured in the temperature range of (328.70 to 499.88) K using the boiling point method. The Antoine equation was used to describe the vapor pressure data, and the parameters in the equation were determined by means of the least-square regression method. The deviation of experimental data from the corresponding calculated values was within 0.30 %. On the basis of the equation, the enthalpy of vaporization of 2-amino-3-methylpyridine at normal boiling point (T_b) was obtained from the slope of the plot of $\ln p$ vs $1/T$. The standard enthalpy of vaporization $\Delta_{\text{vap}}H(298.15 \text{ K})$ was estimated by Othmer's method. Using 3-methylpyridine as the standard substance, the value of $\Delta_{\text{vap}}H(298.15 \text{ K})$ was found to be 62.58 $\text{kJ}\cdot\text{mol}^{-1}$. A parallel value of $\Delta_{\text{vap}}H(298.15 \text{ K})$ was also obtained using benzene as the standard substance. Additionally the Watson relation was employed to verify the value of $\Delta_{\text{vap}}H(298.15 \text{ K})$, from which it was shown that the value of the standard enthalpy of vaporization was acceptable.

Introduction

The vapor pressure data of a pure substance are fundamentally important to phase equilibrium calculations and some other engineering applications, such as distillation, evaporation, and two-phase reactors. Experimental data over a wide temperature range are scarce for the substances with higher boiling points. Besides, the enthalpy of vaporization is also one of the most important thermodynamic parameters for the design and operation of multicomponent and multistage vapor–liquid equilibrium processes. The measurement and correlation of enthalpy of vaporization is valuable to both engineering work and theoretical research.

2-Amino-3-methylpyridine (CASRN 1603-40-3) is used as an intermediate for manufacturing pharmaceuticals.^{1–3} It is usually prepared by the Chichibabin reaction between 3-methylpyridine (CASRN 108-99-6) and sodium amide (CASRN 7782-92-5) in an organic solvent. The product of 2-amino-3-methylpyridine with high purity is obtained by vacuum distillation.^{4,5} Unfortunately, a literature survey revealed that only limited vapor pressure data^{5,6} on this compound were available. In addition, there is no report on the enthalpy of vaporization of 2-amino-3-methylpyridine as far as we know.

In this study, the vapor pressure of 2-amino-3-methylpyridine was measured using the boiling point method at temperatures from (328.70 to 499.88) K. The experimental data of vapor pressure were correlated with the Antoine equation. The constants of the equation were determined by means of the least-square regression method. The vaporization enthalpies at the normal boiling point and 298.15 K were also estimated.

Experimental Section

Materials. 2-Amino-3-methylpyridine was prepared using *o*-xylene as solvent according to the literature.^{4,5} It was purified prior to use by distillation under reduced pressure. Its mass fraction purity, determined by gas chromatography equipped

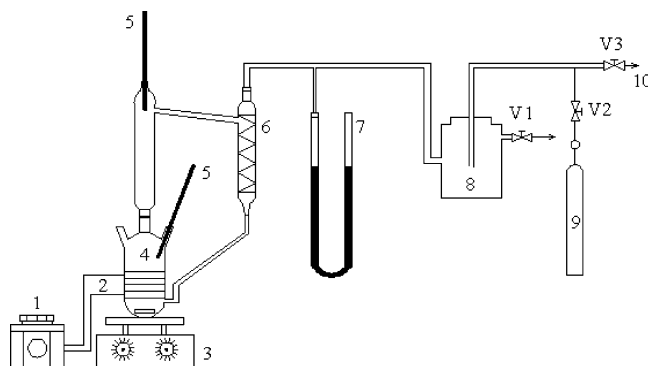


Figure 1. Schematic diagram of the vapor pressure measurement apparatus: 1, variable volt transformer; 2, electric heating tape; 3, magnetic stirrer; 4, boiling chamber; 5, thermometer; 6, condenser; 7, U-tube mercury manometer; 8, buffer vessel; 9, nitrogen cylinder; 10, to vacuum pump; V1, pressure maintaining valve; V2, pressure reducing valve; V3, needle valve.

with a flame ionization detector (FID), was higher than 99.5 %. Water used here was purified by redistillation. All the compounds were degassed by heating and vacuum before being used for measurement.

Apparatus and Procedures. Vapor Pressure. The vapor pressures were measured by the boiling point method.⁷ Figure 1 is a schematic flow diagram of the apparatus setup, and the parts are described below the figure. Briefly, the apparatus consists of a temperature measurement system, a vacuum pump, and a pressure control and measurement system. Nitrogen was introduced into the system at the beginning to remove air and to compensate pressure when the absolute pressure was higher than atmosphere. The temperature was measured with thermometers with an uncertainty of $\pm 0.05 \text{ K}$. The pressure was determined using a McLeod vacuum gauge in the range less than 0.650 kPa with an uncertainty of $\pm 0.003 \text{ kPa}$ and a U-tube mercury manometer in the range of (0.650 to 111.35) kPa, with an uncertainty of $\pm 0.03 \text{ kPa}$. The McLeod vacuum gauge, U-tube mercury manometer, and thermometers were calibrated before experimental measurement.

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Table 1. Experimental Vapor Pressure of Water and Comparison with Literature Data^a

<i>T</i> /K	<i>p</i> _{exptl} /kPa	<i>p</i> _{lit} /kPa	100 $\Delta p/p^a$
297.95	3.14	3.13	-0.32
303.85	4.41	4.42	0.23
309.05	5.93	5.91	-0.34
313.25	7.44	7.42	-0.27
318.75	9.86	9.89	0.30
322.95	12.20	12.22	0.16
329.35	16.71	16.68	-0.18
333.45	20.18	20.21	0.15
338.55	25.51	25.47	-0.16
342.85	30.80	30.77	-0.10
348.25	38.66	38.72	0.16
353.65	48.42	48.34	-0.17
358.35	58.20	58.27	0.12
362.95	69.67	69.58	-0.13
368.45	85.35	85.47	0.14
371.75	96.28	96.37	0.09
373.25	101.56	101.69	0.13
375.75	111.23	111.10	-0.12

^a 100 $\Delta p/p = 100(p_{\text{lit}} - p_{\text{exptl}})/p_{\text{exptl}}$, where p_{lit} is the data reported in the literature⁸ and p_{exptl} is the experimental value.

To check the reliability and accuracy of the apparatus setup, vapor pressure measurements of water were made from (297.95 to 375.75) K and compared with literature values.⁸

The degassed sample with an approximate volume of 150 mL was placed in the vessel. Prior to measurement, dry nitrogen was introduced into the system to remove air to avoid oxidation of the sample. All the measurements were conducted in a sequence of increasing pressure, and the pressure was controlled at the desired value at every experimental point. When the desired pressure was lower than atmosphere, the pressure could be reached and maintained by adjusting the vacuum system. When the desired pressure was higher than atmosphere, the system could be pressurized with dry nitrogen and the pressure kept by the pressure maintaining valve V1. At every desired pressure, the sample was heated and stirred well with a magnetic stirrer to provide isothermal conditions and to prevent superheating. When thermal equilibrium was reached, the temperature and pressure of the system were recorded. The experiment was repeated 3 to 4 times at each pressure, and an averaged value was taken.

Results and Discussion

Vapor Pressure of 2-Amino-3-methylpyridine. The vapor pressures of water were determined from (297.95 to 375.75) K. As shown in Table 1, the deviations between experimental

Table 3. Antoine Constants for 2-Amino-3-methylpyridine and Calculated Average Absolute Deviations

Antoine constants			temperature range	
A	B	C	K	100 AAD
14.8098	4213.632	-82.613	328.70 to 499.88	0.30

results and the literature values were within 0.4 %, which shows the apparatus to be reliable. The deviation is defined as

$$\Delta p/p = \frac{p_{\text{lit}} - p}{p} \quad (1)$$

The vapor pressures of 2-amino-3-methylpyridine were measured in a temperature range from (328.70 to 499.88) K. The results were listed in Table 2. The experimental data were correlated with the Antoine equation⁹

$$\ln(p/\text{kPa}) = A - \frac{B}{T/\text{K} + C} \quad (2)$$

where p is the vapor pressure; T is the absolute temperature; and A , B , and C are Antoine constants. The optimum Antoine constants A , B , and C were determined by a least-square regression method. The values of A , B , and C and the absolute average deviation (AAD) were shown in Table 3. The AAD is defined by

$$\text{AAD}(p) = \frac{1}{N} \sum |p_i - p_{i,\text{calcd}}|/p_i \quad (3)$$

where N is the number of data points; $p_{i,\text{calcd}}$ is the calculated value from eq 2; and p_i is the experimental value.

Figure 2 illustrates the variation of vapor pressure with temperature, where the smoothed line is calculated from the Antoine equation (eq 2). Figure 3 shows the deviation distribution of the data correlations, where the deviation is defined as

$$\Delta p/p = \frac{p_{\text{calcd}} - p}{p} \quad (4)$$

The individual deviations are within ± 0.5 % for a majority of data points. It appears that the Antoine equation represents the data well over temperature.

Abramovitch et al.⁵ reported a boiling temperature range of (373.15 to 376.15) K at a reduced pressure of 1.47 kPa. Buckingham et al.⁶ reported two boiling temperatures, (375.15 to 376.15) K at 1.47 kPa and 495 K at 99.73 kPa, which are in good agreement with the data of this work, 374.67 K at 1.47 kPa and 495.13 K at 99.74 kPa. In addition, the normal boiling

Table 2. Experimental Vapor Pressure Data of 2-Amino-3-methylpyridine and Calculated Deviations

<i>T</i> /K	<i>p</i> _{exptl} /kPa	100 $\Delta p/p^a$	<i>T</i> /K	<i>p</i> _{exptl} /kPa	100 $\Delta p/p^a$	<i>T</i> /K	<i>p</i> _{exptl} /kPa	100 $\Delta p/p^a$
328.70	0.098	1.00	411.35	7.34	-0.12	455.25	33.15	0.11
329.84	0.108	-0.82	415.63	8.67	-0.30	456.78	34.61	0.43
336.23	0.165	-0.26	421.50	10.75	0.11	457.95	36.08	-0.22
344.13	0.271	0.31	425.85	12.61	-0.09	460.15	38.48	-0.12
349.18	0.368	0.24	428.75	13.95	0.10	461.72	40.22	0.08
354.89	0.515	-0.21	431.43	15.29	0.28	469.63	50.38	0.27
357.64	0.598	0.31	435.66	17.74	-0.11	478.22	63.87	0.19
359.93	0.69	-1.34	439.42	20.12	-0.12	484.84	76.42	-0.22
363.55	0.82	0.98	443.51	23.01	-0.16	490.45	87.65	0.48
368.61	1.08	-0.03	447.94	26.50	-0.13	495.13	99.74	-0.72
374.67	1.47	-0.29	448.95	27.30	0.08	496.05	101.44	-0.14
387.23	2.67	-0.49	450.76	28.88	0.11	497.73	105.38	0.17
390.85	3.13	-0.14	451.84	29.81	0.29	498.8	108.53	-0.17
398.46	4.32	0.58	454.19	32.09	0.14	499.88	111.35	-0.11
405.57	5.82	0.14						

^a 100 $\Delta p/p = 100(p_{\text{calcd}} - p_{\text{exptl}})/p_{\text{exptl}}$, where p_{calcd} was calculated from the Antoine equation and p_{exptl} is the experimental value.

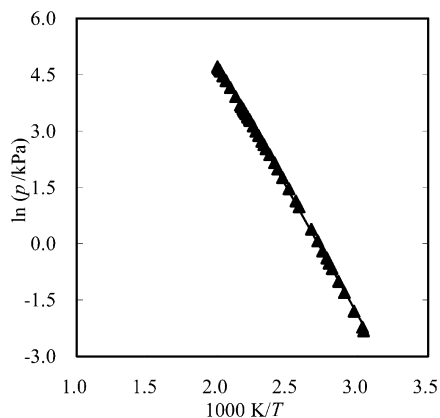


Figure 2. Temperature dependence of vapor pressures for 2-amino-3-methylpyridine: $\blacktriangle$, experimental data; solid line, calculated by eq 2.

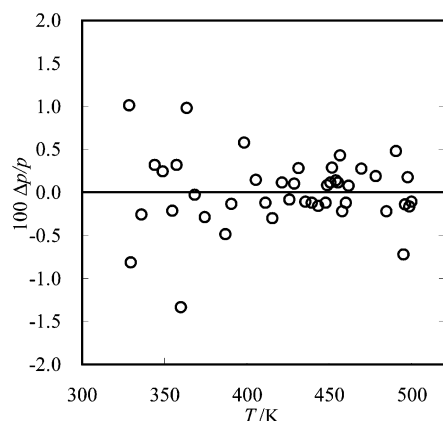


Figure 3. Deviation distributions of the calculated values of the Antoine equation from the experimental vapor pressures for 2-amino-3-methylpyridine.

temperature calculated from eq 2 was 496.06 K which was used as the value of T_b for 2-amino-3-methylpyridine hereinafter.

$\Delta_{\text{vap}}H(T_b)$ for 2-Amino-3-methylpyridine. When the vapor phase of a pure fluid is in equilibrium with its liquid phase, the equality of chemical potential, temperature, and pressure in both phases leads to the Clausius–Clapeyron equation¹⁰

$$\frac{dp}{dT} = \frac{\Delta_{\text{vap}}H}{T(V_G - V_L)} = \frac{\Delta_{\text{vap}}H}{(RT^2/p)\Delta Z_v} \quad (5)$$

where $\Delta Z_v = Z - Z_L$ is the difference between the compressibility factors of the vapor and liquid; p is the vapor pressure; T is the absolute temperature; $\Delta_{\text{vap}}H$ is the phase change enthalpy; V_G is the vapor volume; and V_L is the liquid volume.

The Clausius–Clapeyron equation is a general equation originally relating vapor pressure, temperature, volume change, and enthalpy of vaporization of a pure liquid in equilibrium with the gas phase. If the volume of the liquid is much smaller than that of the gas and therefore is neglected and the gas-phase behavior is treated as ideal, then this equation becomes¹¹

$$\frac{d \ln(p)}{d(1/T)} = \frac{-\Delta_{\text{vap}}H}{R} \quad (6)$$

where $\Delta_{\text{vap}}H$ is the enthalpy of vaporization and R is the gas constant.

Accurate vapor pressure values can be used to reliably estimate the enthalpy of vaporization.¹² Taking eq 2 into eq 6, the temperature dependency of the enthalpy of vaporization can be estimated according to the equation

$$\Delta_{\text{vap}}H(T) = \frac{BRT^2}{(T + C)^2} \quad (7)$$

where B and C are the Antoine constants.

Equation 7 can be used to calculate the value of the enthalpy of vaporization at the normal boiling point ($\Delta_{\text{vap}}H(T_b)$). On the basis of the values of Antoine constants shown in Table 3, the value of $\Delta_{\text{vap}}H(T_b)$ for 2-amino-3-methylpyridine was determined to be 50.43 kJ·mol⁻¹.

Estimation of $\Delta_{\text{vap}}H(298.15 \text{ K})$ for 2-Amino-3-methylpyridine. Because eq 7 is valid in the temperature range of (328.66 to 499.88) K, the standard enthalpy of vaporization $\Delta_{\text{vap}}H(298.15 \text{ K})$ of 2-amino-3-methylpyridine at 298.15 K cannot be calculated directly from the equation. Fortunately, Othmer's method can be used to estimate the value of $\Delta_{\text{vap}}H(298.15 \text{ K})$ as follows.¹³

Equation 6 can be rearranged to yield

$$\frac{1}{\Delta_{\text{vap}}H} \cdot \frac{dp}{p} = \frac{dT}{RT^2} \quad (8)$$

Equation 8 refers to any substance consistent with the assumption that vapor phase is an ideal gas, and the molar volume of liquid is negligible compared to the molar volume of vapor. The same equation applies to any other consistent material at the same temperature

$$\frac{1}{\Delta_{\text{vap}}H'} \cdot \frac{dp'}{p'} = \frac{dT}{RT^2} \quad (9)$$

where p' represents the vapor pressures of the second substance at temperature T , and $\Delta_{\text{vap}}H'$ represents the vaporization enthalpy of the second substance also at temperature T .

Because the right-hand side of the equation will be the same for any substance at the same temperature, combining eq 8 and eq 9 gives the following

$$\frac{dp/p}{dp'/p'} = \frac{\Delta_{\text{vap}}H}{\Delta_{\text{vap}}H'} \text{ or } \frac{d \log p}{d \log p'} = \frac{\Delta_{\text{vap}}H}{\Delta_{\text{vap}}H'} \quad (10)$$

These equations indicate that, if the differential of the pressure for one substance divided by the mean vapor pressure of that substance is plotted against the differential of the pressure of the second substance at the same temperature divided by the vapor pressure at that temperature, a straight line should be obtained, and the slope of the straight line will be the ratio of the vaporization enthalpy of these two substances at the same temperature. The second substance is usually referred to as a standard substance, whose enthalpy of vaporization at 298.15 K and Antoine constants are available.

Herein, a standard substance should be chemically similar to the substance under investigation. 3-Methylpyridine was chosen as the standard compound because its vapor pressure and Antoine constants¹⁴ are accurately known, and its standard enthalpy of vaporization at 298.15 K was reported to be 44.57 kJ·mol⁻¹.¹⁵ Figure 4 showed the plot of $\log p$ of 2-amino-3-methylpyridine against that ($\log p'$) of 3-methylpyridine, which is approximately a straight line (line 1). Using the least-square method, we could determine the slope ($\Delta_{\text{vap}}H/\Delta_{\text{vap}}H'$) of the line to be 1.404, and the regressive equation was as follows

$$\log p = 1.404 \log p' - 1.8543 \quad (11)$$

On the basis of eq 11, the value of the enthalpy of vaporization $\Delta_{\text{vap}}H(298.15 \text{ K})$ for 2-amino-3-methylpyridine is 62.58 kJ·mol⁻¹.

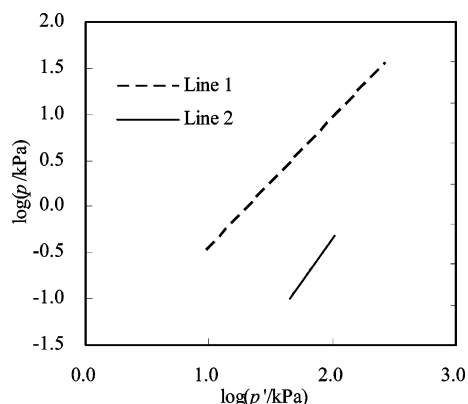


Figure 4. Integral plot of 2-amino-3-methylpyridine against the standard compound: line 1, 3-methylpyridine; line 2, benzene.

To validate the value of $\Delta_{\text{vap}}H(298.15 \text{ K})$, another substance, benzene, was also chosen as the standard substance, whose $\Delta_{\text{vap}}H'(298.15 \text{ K})$ was reported to be $33.92 \text{ kJ}\cdot\text{mol}^{-1}$,¹⁵ and its Antoine constants are also reported in the literature.¹⁶ The plot of $\log p$ against $\log p'$ was shown in Figure 4 (line 2), and the regressive equation was as follows

$$\log p = 1.886 \log p' - 4.1116 \quad (12)$$

So the enthalpy of vaporization $\Delta_{\text{vap}}H(298.15 \text{ K})$ for 2-amino-3-methylpyridine derived using benzene is $63.97 \text{ kJ}\cdot\text{mol}^{-1}$, which is close to that obtained with 3-methylpyridine ($62.58 \text{ kJ}\cdot\text{mol}^{-1}$).

Verification of $\Delta_{\text{vap}}H(298.15 \text{ K})$ for 2-Amino-3-methylpyridine. The enthalpy of vaporization at the normal boiling point $\Delta_{\text{vap}}H(T_b)$ can be extended to a desired temperature, using the well-known relation reported by Watson.¹⁷ To further verify the reliability of Othmer's method, the value of $\Delta_{\text{vap}}H(298.15 \text{ K})$ for 2-amino-3-methylpyridine can be estimated independently according to the value of $\Delta_{\text{vap}}H(T_b)$.

The Watson relation used to estimate $\Delta_{\text{vap}}H(T)$ at any temperature for a pure substance is as follows

$$\Delta_{\text{vap}}H(T) = \Delta_{\text{vap}}H(T_b) \left(\frac{1 - T/T_c}{1 - T_b/T_c} \right)^n \quad (13)$$

where T_c is the critical temperature; T_b is the normal boiling point; and $\Delta_{\text{vap}}H(T_b)$ is the enthalpy of vaporization at T_b .

Viswanath and Kuloor¹⁸ recommended that n in eq 13 can be obtained by the expression

$$n = \left(0.00264 \frac{\Delta_{\text{vap}}H(T_b)}{RT_b} + 0.8794 \right)^{10} \quad (14)$$

The boiling point of 2-amino-3-methylpyridine is 496.06 K . Taking T_b and $\Delta_{\text{vap}}H(T_b)$ to eq 14, n is calculated to be 0.3967 .

To estimate the $\Delta_{\text{vap}}H(298.15 \text{ K})$ of 2-amino-3-methylpyridine, the critical temperature of 2-amino-3-methylpyridine must be known. However, no data on this compound are available. T_c can be estimated by the equation proposed by Riedel et al.:¹⁰ $T_c = T_b/\theta$. In this study, the Lydersen method¹⁹ was used to estimate θ

$$\theta = 0.567 + \sum \Delta_T - (\sum \Delta_T)^2 \quad (15)$$

where $\sum \Delta_T$ was obtained by adding the contributions listed in Table 4.

Taking $\sum \Delta_T$ to eq 15, θ could be estimated to be 0.667 . Finally, T_c was calculated to be 743.72 K . Taking T_b , T_c , and

Table 4. Structural Contributions to Calculate Δ_T in Equation 15¹⁰

group	contribution number	Δ_T
=CH (in ring)	3	0.011^{18}
=C (in ring)	2	0.011^{18}
>N (in ring)	1	0.007^{18}
—CH_3	1	0.020^{18}
—NH_2	1	0.031^{18}
$\sum \Delta_T$		0.113

$\Delta_{\text{vap}}H(T_b)$ into eq 13, the enthalpy of vaporization of 2-amino-3-methylpyridine can be calculated at any selected temperature. Finally, taking $T = 298.15 \text{ K}$ to eq 13, $\Delta_{\text{vap}}H(298.15 \text{ K})$ was calculated to be $63.66 \text{ kJ}\cdot\text{mol}^{-1}$, which is well in agreement with the values obtained from Othmer's method. The deviations were 1.72% and -0.48% for 3-methylpyridine and benzene as standard substances, respectively.

Conclusion

The vapor pressures of 2-amino-3-methylpyridine were determined in the temperature range from $(328.70 \text{ to } 499.88) \text{ K}$ using the boiling point method. The data were represented by the Antoine equation with an absolute average deviation of 0.30% . On the basis of the values of Antoine constants, the $\Delta_{\text{vap}}H(T_b)$ for 2-amino-3-methylpyridine was calculated by the Clausius–Clapeyron equation. The $\Delta_{\text{vap}}H(298.15 \text{ K})$ for 2-amino-3-methylpyridine was also estimated by Othmer's method using 3-methylpyridine and benzene as the standard substance, respectively. The results showed that there was a small deviation between the estimated values and those values derived from different standard substances.

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