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## CHEMICAL BIOLOGICAL CENTER

U.S. ARMY RESEARCH, DEVELOPMENT AND ENGINEERING COMMAND

ECBC-TR-480

### VAPOR PRESSURE OF RUSSIAN VX

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U.S. Army Edgewood Chemical Biological Center  
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| 14. ABSTRACT<br>O-isobutyl-S-(diethylaminoethyl) methyl phosphonothiolate (RVX) is a highly toxic compound and a structural isomer of the nerve agent, O-ethyl-S-(diisopropylaminoethyl) methyl phosphonothiolate (VX). The vapor pressure of RVX has been measured using a modified ASTM saturation method between -10 and 18 °C and a second ASTM method employing differential thermal analysis between 145 and 232 °C. Those data and an Antoine fit to the data are presented and compared to an earlier correlation based on data measured over a narrower temperature range.                                                                                                                                                                                                                                                                                                                                                                |             |                         |                               |                                                         |                                                                |
| 15. SUBJECT TERMS<br>O-isobutyl-S-(diethylaminoethyl) methyl phosphonothiolate<br>Differential Thermal Analysis<br>Gas Chromatography<br>DTA<br>RVX<br>Antoine constants<br>Gas Saturation<br>VX<br>Vapor Pressure<br>Russian VX<br>Volatility                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |             |                         |                               |                                                         |                                                                |
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## PREFACE

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# VAPOR PRESSURE OF RUSSIAN VX

## 1. INTRODUCTION

Russian VX, O-isobutyl-S-(diethylaminoethyl) methyl phosphonothiolate (RVX)<sup>1-3</sup>, is a highly toxic compound and a structural isomer of the nerve agent, O-ethyl-S-(diisopropylaminoethyl) methyl phosphonothiolate (VX). The chemical structures of VX and RVX are illustrated in Figure 1.

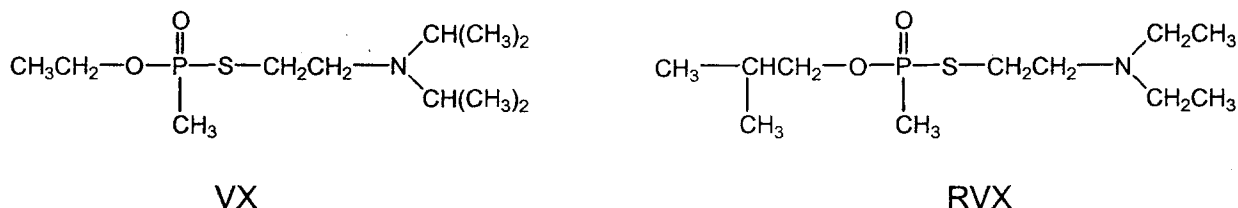


Figure 1. Chemical Structures for VX and RVX.

Vapor pressure data for toxic materials such as chemical warfare agents are useful in predicting downwind concentration dynamics following dissemination. The data are also useful in determining system designs for generating and precisely controlling vapor concentrations for toxicology investigations and detector calibration. Recent studies from this Laboratory (ECBC, APG, MD) have reported the vapor pressure of VX between -13 and 20 °C using a vapor saturation method and have analyzed that data in the light of historical VX data.<sup>4</sup> The vapor pressure of RVX has been measured in this work between -10 and 18 °C using vapor saturation,<sup>5</sup> and between 145 and 232 °C using differential thermal analysis (DTA).<sup>6</sup> The current work compares favorably to a correlation published recently by Rittfeldt<sup>3</sup> based on measured data between -10 and 103 °C and significantly extends the range of measured data on the high end.

## 2. EXPERIMENTAL METHODS

The experimental methods used in this study to generate the saturator data are identical to those used previously to measure VX vapor pressure between -13 and 20 °C<sup>4</sup> and will be briefly described here. Vapor streams saturated with RVX were generated by flowing nitrogen carrier gas at 30 standard cubic centimeters per minute (sccm) through a glass vessel, i.e., saturator, containing liquid RVX. In this work, the saturator was loaded with 5 g of liquid RVX, which was analyzed before vapor pressure data were measured using Nuclear Magnetic Resonance (NMR) Spectroscopy (<sup>31</sup>P, <sup>13</sup>C, and <sup>1</sup>H). NMR analysis of the RVX liquid sample used in the saturator work indicated 71.9-mole% purity at the beginning of the vapor pressure experiment. The liquid RVX sample purity did not change after 2 weeks of vapor pressure measurement.

The carrier gas used in the present work was ultra-high purity (UHP) grade nitrogen, and the saturator effluent was sampled as shown in Figure 2 by drawing 20 sccm, i.e., corrected to 0 °C and 1 atmosphere, to a modified ACEM Model 900 (Dynatherm Analytical Instruments Inc., Kelton, PA) adsorbent (tenax) concentrator collection tube for between 1 and 40 min. The total volume of RVX vapor sampled ranged from 20 to 800 standard cc. After sample collection, the tenax collection tube was rapidly heated to 275 °C under a flow rate of 20-sccm UHP-grade helium for 4 min and transferred to the ACEM 900 tenax focusing trap maintained at 35 °C. Transfer continued for 1 additional min to allow the 10-mm o.d. tenax collection tube to cool. Then, the focusing trap was rapidly heated to 300 °C under a flow rate of 9.3-sccm UHP grade helium for 3 min to ensure sample transfer to the gas chromatographic (GC) column. The 30-m x 0.53-mm i.d. fused silica GC column (Restek Corp., Bellefonte, PA), with a 1.0- $\mu$ m RTx-1 (polydimethylsiloxane) stationary phase, was maintained at 50 °C for 2 min following sample introduction, then heated at a rate of 10 °C/min to 200 °C, and then maintained at 200 °C for 5 min, well after the RVX eluted. A Hewlett-Packard Model 5890 Series II GC equipped with a flame-photometric detector operating in the phosphorus mode (FPDP) was used as the detection system in this work. Helium was used as the GC carrier gas at a flow rate of 9.3 cc/min and as detector make-up gas at a flow rate of 10.7 cc/min. The combustion gases were air (100 cc/min) and hydrogen (75 cc/min). Using the instrumentation and operating conditions described, RVX eluted at 15.7 min, which corresponds to a GC column temperature of 187 °C. Initial saturator GC analyses revealed about 30 significant phosphorus-containing compounds. The peak identified as RVX initially accounted for about 11% of the total area under the peaks as opposed to the calibration data which showed that the major peak RVX accounted for about 80% of the total area, consistent with the fact that the FPDP is only sensitive to the analyte and phosphorus-bearing impurities. The prominence of the RVX GC peak remained relatively unchanged during vapor pressure data measurement.

The FPDP response to RVX was calibrated as before for VX.<sup>4</sup> The response observed is described by the following equation:

$$y = ax^2 + bx$$

where

$$\begin{aligned} y &= \text{GC area (10}^6 \text{ area counts)} \\ x &= \text{analyte mass (ng)} \\ a &= 0.001015 \\ b &= 0.3796 \end{aligned}$$

Calculation of vapor pressure from the RVX mass, indicated by the area of the FPDP signal at each measurement temperature, was performed as before.<sup>4</sup> The vapor pressure was calculated at each temperature as before, using the sample purity to correct the indicated vapor pressure by dividing the RVX mole fraction in accordance with Raoult's Law.

DTA is a useful technique for measuring vapor pressure in the 100 to 10,000 Pa range and is well documented in literature.<sup>7-10</sup> DTA was used in the present work to measure the vapor pressure of RVX between 145 and 232 °C. The purity of the sample used for DTA measurements was determined to be 92.7 weight% by NMR. No purity correction has been applied to the DTA data.

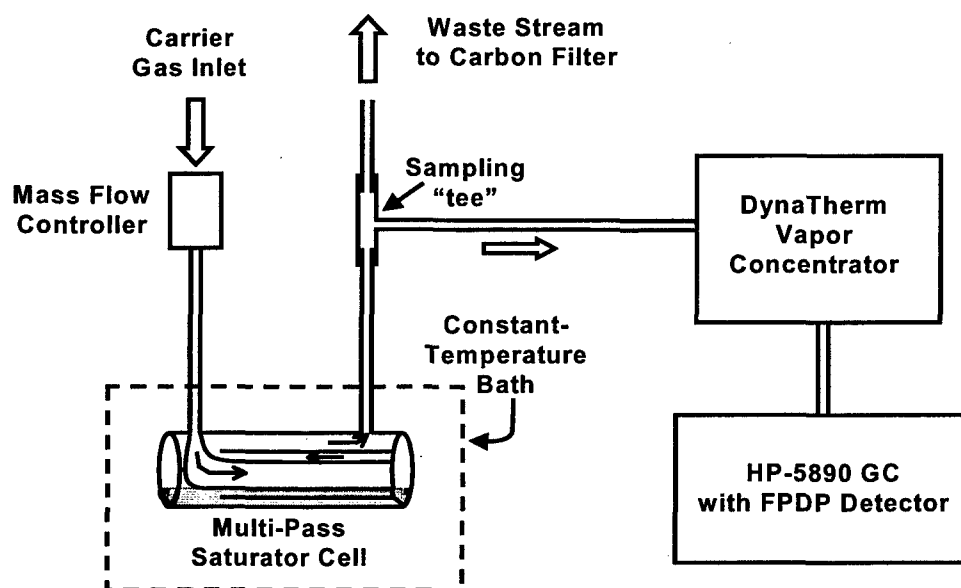


Figure 2. Schematic of Data Acquisition System Used to Measure RVX Vapor Pressure.

### 3. RESULTS

Initial saturator effluent GC chromatograms revealed the presence of approximately 30-major components, with RVX accounting for about 11% of the total GC area. This observation is similar to previous observations for VX<sup>4</sup> and is exacerbated by the low volatility of RVX compared to its impurities. Since the FPDP is selective for phosphorus-containing materials, this measurement represents an upper limit estimate of the mole percentage of RVX vapor in the effluent. The indicated percentage of RVX in the saturator effluent stream at the end of data measurement increased slightly to 12% of the total phosphorus compound area, presumably due to selective evaporation of lighter impurities during the data measurement process. This change is consistent with a slight increase in liquid phase purity, but not enough to affect the data analysis. Data measured in this work for RVX are shown in Figure 3 along with the RVX Antoine Equation derived from the new data.

Table 1 shows the RVX vapor pressures resulting from 7 separate saturator determinations at temperatures between -10 and 18 °C. Each measurement listed in Table 1 in this temperature range is actually the average of between 5 and 15 separate measurements taken after the data acquisition system had stabilized at each

new condition. As observed before for VX, it took several hours to establish steady state conditions following each temperature change. One experiment was performed at a saturator flow rate of 50 sccm. The measured vapor pressure of RVX did not change with saturator flow rate, confirming that equilibrium had been achieved at both flow rates. All saturator data listed in Table 1 were measured using a saturator flow rate of 30 sccm and a sample flow rate of 20 sccm.

Table 1. Vapor Pressure for RVX. (Measured using the saturator (-10 to 18 °C) and DTA (145.6 to 232 °C) methods, and calculated using Antoine Correlation of all data and percent difference.)

| Temp (°C) | Measured RVX Vapor Pressure (Pa) | Calculated RVX Vapor Pressure (Pa) | Difference (%) |
|-----------|----------------------------------|------------------------------------|----------------|
| -10.0     | $9.54 \times 10^{-4}$            | $9.52 \times 10^{-4}$              | 0.2            |
| -2.5      | $2.81 \times 10^{-3}$            | $2.81 \times 10^{-3}$              | 0.0            |
| 5.0       | $7.46 \times 10^{-3}$            | $7.71 \times 10^{-3}$              | 3.3            |
| 11.8      | $1.76 \times 10^{-2}$            | $1.82 \times 10^{-2}$              | 3.0            |
| 14.0      | $2.38 \times 10^{-2}$            | $2.38 \times 10^{-2}$              | 0.0            |
| 16.0      | $3.10 \times 10^{-2}$            | $3.02 \times 10^{-2}$              | 2.8            |
| 18.1      | $3.91 \times 10^{-2}$            | $3.86 \times 10^{-2}$              | 1.3            |
| 145.6     | $5.600 \times 10^2$              | $5.742 \times 10^2$                | 2.5            |
| 154.2     | $7.599 \times 10^2$              | $8.661 \times 10^2$                | 12.3           |
| 156.0     | $8.533 \times 10^2$              | $9.416 \times 10^2$                | 9.4            |
| 158.6     | $1.040 \times 10^3$              | $1.061 \times 10^3$                | 2.0            |
| 162.3     | $1.333 \times 10^3$              | $1.254 \times 10^3$                | 6.3            |
| 166.8     | $1.747 \times 10^3$              | $1.530 \times 10^3$                | 14.2           |
| 169.1     | $9.066 \times 10^2$              | $1.691 \times 10^3$                | 46.4           |
| 174.9     | $2.253 \times 10^3$              | $2.164 \times 10^3$                | 4.1            |
| 181.0     | $2.893 \times 10^3$              | $2.783 \times 10^3$                | 4.0            |
| 187.7     | $3.746 \times 10^3$              | $3.637 \times 10^3$                | 3.0            |
| 198.4     | $5.573 \times 10^3$              | $5.478 \times 10^3$                | 1.7            |
| 209.0     | $7.959 \times 10^3$              | $8.055 \times 10^3$                | 1.2            |
| 221.9     | $1.256 \times 10^4$              | $1.256 \times 10^4$                | 0.0            |
| 232.0     | $1.460 \times 10^4$              | $1.747 \times 10^4$                | 16.4           |

Table 1 also lists the vapor pressure data measured by the DTA method between 145 and 232 °C. As can be easily seen in this Table and Figure 3, the data point at 169.1 °C / 907 Pa is clearly inconsistent with the other DTA data; however, the reason for this discrepancy is unclear, and so this data point has been included in the subsequent data analysis. RVX vapor pressure data measured in this work along with the Antoine Correlation of the data are plotted in Figure 3. Figure 4 is a plot comparing the Antoine Equation, derived by using high- and low-temperature data, with the 2-parameter correlation proposed by Rittfeldt.

#### 4. DISCUSSION

The agreement between the data measured in this work and that reported earlier by Rittfeldt is excellent between 10 and 100 °C. However, at temperatures lower than 10 °C, the Rittfeldt Vapor Pressure Equation predicts values increasing to as much as 30% higher than data measured in this work. The ability to precisely control sub-ambient temperatures and to fully equilibrate the vapor source in the method used by Rittfeldt appears to be questionable and might account for any differences seen in the two data sets below 10 °C.

This study does not include in its correlation the limited-distribution data measured between 30 and 50 °C over 35 years ago for RVX using Knudsen Effusion. Those data are significantly (ca. 35%) lower than predicted based on the work presented in this study and that of Rittfeldt. These data (RVX Knudsen Effusion) are similar to the VX Knudsen data and are only mentioned for historical purposes. The Knudsen data suggest that unaccounted pressure gradients and/or impurity effects could present systematic difficulties for very low volatility materials undergoing this type of measurement.

The Antoine Correlation performed, using only the new data reported in the present work, is shown in Figure 3 and parallels that of VX at an average value of about 72% of VX in the low-temperature range to about 90% at the high end of the range.

The average difference between calculated and observed data for all of the current RVX data is 6.4%. If the questionable DTA data point is excluded, the average difference is reduced to less than 4.4%, which is comparable to other recent results from this Laboratory and considered to represent very good precision in the data measurement. Table 2 lists calculated vapor pressures, volatility, and heats of vaporization for RVX at selected intervals over the range of -20 to 260 °C based on the present data. The boiling point projected for RVX based on the present work is 295 °C.

It is of considerable interest to note the excellent agreement between the 25 °C volatility prediction based on the present work ( $9.06 \text{ mg/m}^3$ ) and that presented by Rittfeldt<sup>3</sup> ( $8.9 \text{ mg/m}^3$ ).

#### 5. CONCLUSIONS

RVX vapor pressure has been measured using the saturator and DTA methods. An Antoine analysis has been performed using the data reported herein. The vapor pressure of RVX is about 72% of VX at -10 °C and rises to 90% of VX at 230 °C. This observation is somewhat at odds with that made by Rittfeldt, whose VX and RVX data seem to converge within the low temperature range (20% difference at 0 °C and 32% difference at 100 °C). However, the current data are in good agreement with those of Rittfeldt between the 10 to 100 °C range.

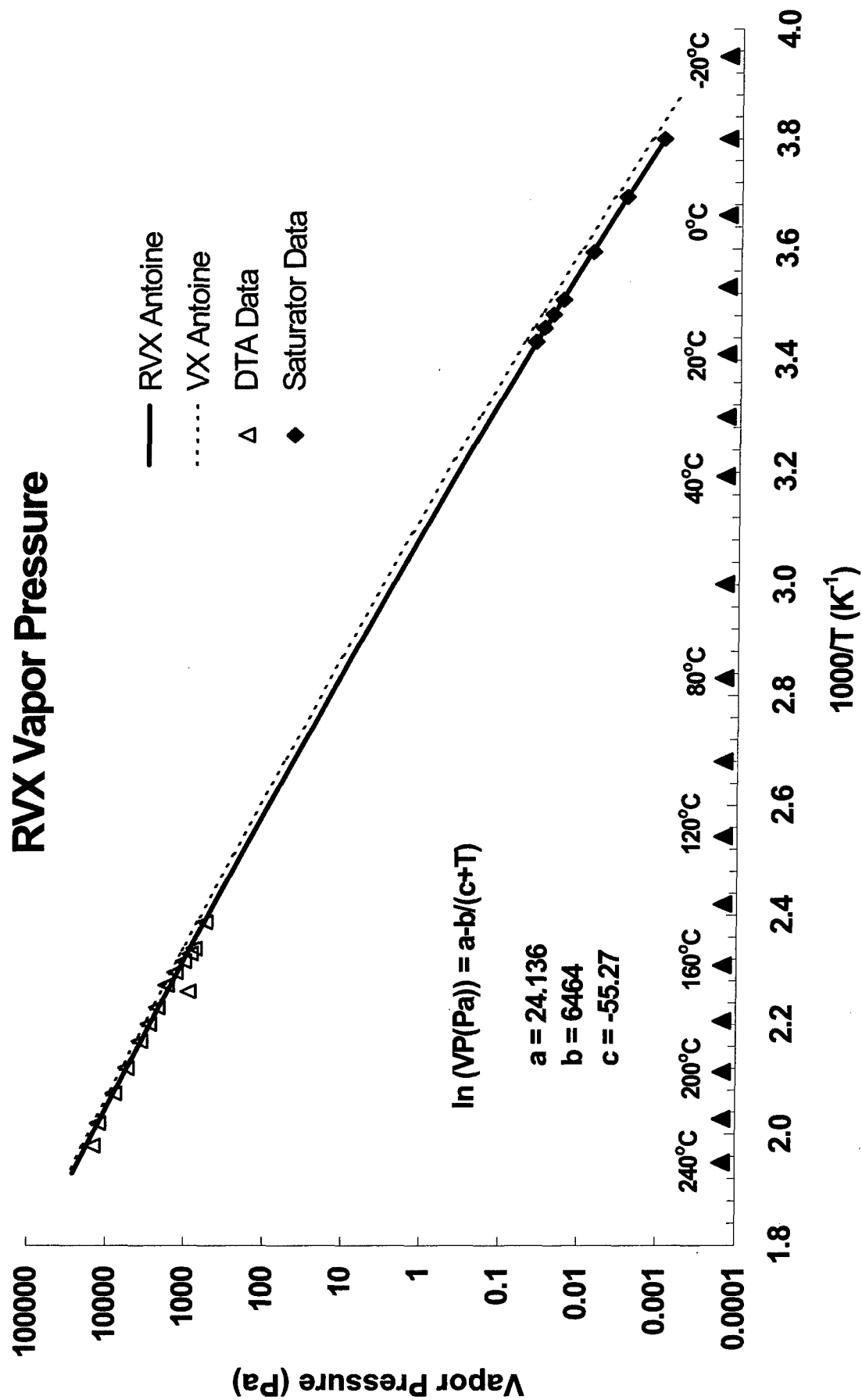


Figure 3. Vapor Pressure Data for RVX between -10 and 232 °C along with Antoine Fits for RVX and VX. (Present DTA and saturator data were combined to obtain the RVX Antoine Fit.)

# RVX Vapor Pressure

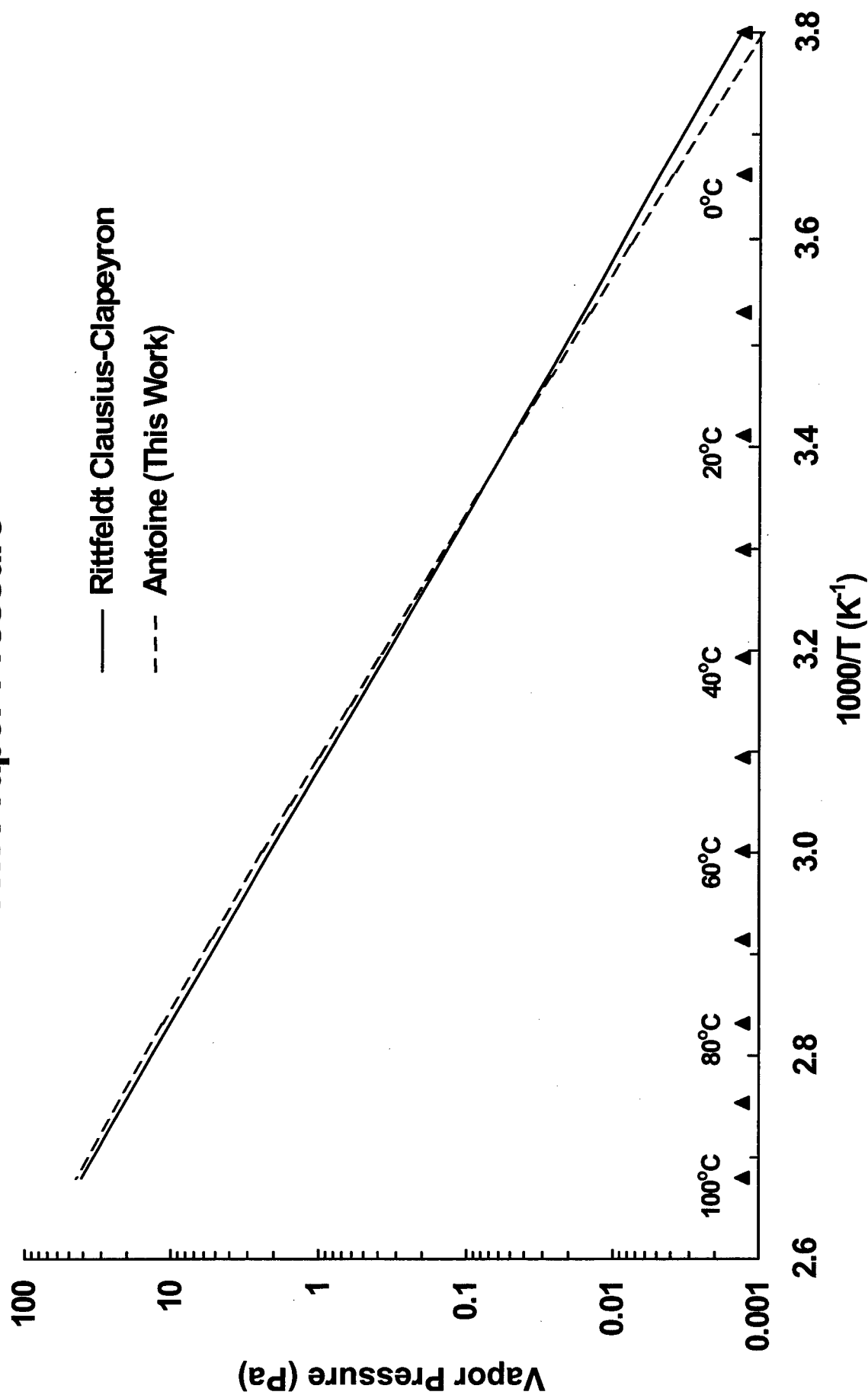


Figure 4. Comparison of Antoine Equation Derived from Current Data and Clausius-Clapeyron Correlation from Rittfeldt.<sup>3</sup>

Table 2. Calculated Vapor Pressures, Volatility, and Heats of Vaporization for RVX Based on Antoine Coefficients Listed in Figure 3. (Bold figures are within the range of measured data.)

| Temp<br>(°C) | Calculated RVX<br>Vapor Pressure (Pa)   | RVX Volatility<br>(mg/m <sup>3</sup> )  | $\Delta H_{\text{vap}}$<br>(kJ/mol) |
|--------------|-----------------------------------------|-----------------------------------------|-------------------------------------|
| -20          | $1.98 \times 10^{-4}$                   | $2.51 \times 10^{-2}$                   | 88.0                                |
| -15          | $4.42 \times 10^{-4}$                   | $5.51 \times 10^{-2}$                   | 87.0                                |
| <b>-10</b>   | <b><math>9.52 \times 10^{-4}</math></b> | <b><math>1.16 \times 10^{-1}</math></b> | <b>86.1</b>                         |
| <b>-5</b>    | <b><math>1.98 \times 10^{-3}</math></b> | <b><math>2.37 \times 10^{-1}</math></b> | <b>85.3</b>                         |
| <b>0</b>     | <b><math>3.96 \times 10^{-3}</math></b> | <b><math>4.67 \times 10^{-1}</math></b> | <b>84.5</b>                         |
| <b>5</b>     | <b><math>7.71 \times 10^{-3}</math></b> | <b><math>8.92 \times 10^{-1}</math></b> | <b>83.7</b>                         |
| <b>10</b>    | <b><math>1.46 \times 10^{-2}</math></b> | <b><math>1.66 \times 10^0</math></b>    | <b>83.0</b>                         |
| <b>15</b>    | <b><math>2.68 \times 10^{-2}</math></b> | <b><math>2.99 \times 10^0</math></b>    | <b>82.3</b>                         |
| <b>20</b>    | <b><math>4.80 \times 10^{-2}</math></b> | <b><math>5.27 \times 10^0</math></b>    | <b>81.6</b>                         |
| 25           | $8.40 \times 10^{-2}$                   | $9.06 \times 10^0$                      | 81.0                                |
| 30           | $1.44 \times 10^{-1}$                   | $1.52 \times 10^1$                      | 80.4                                |
| 35           | $2.41 \times 10^{-1}$                   | $2.51 \times 10^1$                      | 79.8                                |
| 40           | $3.95 \times 10^{-1}$                   | $4.06 \times 10^1$                      | 79.2                                |
| 45           | $6.36 \times 10^{-1}$                   | $6.43 \times 10^1$                      | 78.7                                |
| 50           | $1.01 \times 10^0$                      | $1.00 \times 10^2$                      | 78.2                                |
| 55           | $1.57 \times 10^0$                      | $1.54 \times 10^2$                      | 77.7                                |
| 60           | $2.40 \times 10^0$                      | $2.32 \times 10^2$                      | 77.2                                |
| 70           | $5.38 \times 10^0$                      | $5.04 \times 10^2$                      | 76.4                                |
| 80           | $1.14 \times 10^1$                      | $1.04 \times 10^3$                      | 75.5                                |
| 100          | $4.48 \times 10^1$                      | $3.86 \times 10^3$                      | 74.1                                |
| 120          | $1.49 \times 10^2$                      | $1.22 \times 10^4$                      | 72.8                                |
| 140          | $4.35 \times 10^2$                      | $3.38 \times 10^4$                      | 71.6                                |
| <b>160</b>   | <b><math>1.13 \times 10^3</math></b>    | <b><math>8.40 \times 10^4</math></b>    | <b>70.6</b>                         |
| <b>180</b>   | <b><math>2.67 \times 10^3</math></b>    | <b><math>1.90 \times 10^5</math></b>    | <b>69.7</b>                         |
| <b>200</b>   | <b><math>5.81 \times 10^3</math></b>    | <b><math>3.95 \times 10^5</math></b>    | <b>68.9</b>                         |
| <b>220</b>   | <b><math>1.18 \times 10^4</math></b>    | <b><math>7.68 \times 10^5</math></b>    | <b>68.2</b>                         |
| 240          | $2.25 \times 10^4$                      | $1.41 \times 10^6$                      | 67.5                                |
| 260          | $4.05 \times 10^4$                      | $2.45 \times 10^6$                      | 66.9                                |

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