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EDGEWOOD ARSENAL TECHNICAL REPORT

EC-TR-76058

THE VAPOR PRESSURE OF CHEMICAL AGENTS
GD, VX, EA 2223, EA 3547, EA 3580, EA 5365, AND EA 5533

by

James J. Savage, Ph.D.
Donald Fielder

Chemical Laboratory

August 1976

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Vapor pressure equations valid over an extended temperature and pressure range are presented in parametric, graphical, and tabular form for seven chemical agents: GD, VX, EA 2223, EA 3547, EA 3580, EA 5365, and EA 5533. The Antoine vapor pressure equation and the precision which the experimental data fit this equation were derived by computer analysis. The calculated heats of vaporization and volatilities are listed at selected temperatures for each compound. The chemical purities and techniques used to measure the vapor pressure of each compound are also cited in this report.		

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I. INTRODUCTION.

Vapor pressure data on chemical agents are usually reported over different temperature and pressure ranges by different techniques and workers. Extrapolation of available data from different sources sometimes yields different values at a specified temperature and pressure. An attempt to choose the "best" value is sometimes difficult since sample purity and accuracy of technique are often not stated.

The purpose of this research was to measure the vapor pressure of standard and candidate chemical agents between 1 micron (10^{-3} torr) and approximately 1 torr and combine these data with other data obtained at higher temperatures and pressures. In addition, the vapor pressure of GD was also measured between 1 torr and 25 torr with an isoteniscope.¹ The new work being reported herein comprised vapor pressure measurements obtained at temperatures below 100°C by a static method using an isoteniscope¹ and the Knudsen effusion method.² These data were then consolidated with data previously obtained at higher temperatures, as previously reported,³⁻⁵ and a best fit predictive Antoine equation for the combined data was obtained for each compound. The chemical purity of each compound studied was obtained and recorded together with the method of analysis.

II. EXPERIMENTATION.

An isoteniscope¹ was used in the static method of measuring vapor pressure. It consisted of a Pyrex glass reservoir sealed to a glass U-tube which was immersed in a constant temperature bath and connected to a manometer and adjustable vacuum system. The sample was loaded into the reservoir, degassed, and a portion of the sample was allowed to enter the U-tube by tilting the isoteniscope. The pressure on the vacuum side of the U-tube was adjusted to equalize the height of the sample levels in each arm of the U-tube. A cathetometer was used to determine when the liquid levels were the same. Thus, the pressure on the reservoir side of the U-tube due to the vapor pressure of the sample at the bath temperature was equal to the pressure on the vacuum side. This pressure was then read directly from a calibrated Wallace and Tiernan model FA187 manometer. Calibration was performed by measuring the vapor pressure of ACS reagent-grade n-decane from 30° to 90°C. The results obtained agreed with literature data⁶ to within $\pm 3\%$. This method was used to measure vapor pressures above 1 torr.

In the Knudsen effusion method,² a Cahn model RG electrobalance was used to record weight loss versus time. The sample was suspended from one arm of the balance in a gold-lined magnesium cup. The cover of the cup was made of tantalum foil having a small orifice ($\sim 10^{-4}$ cm²). The vapor pressure of the sample was determined by recording weight loss from the cup versus time at a controlled temperature under reduced pressure (10^{-7} torr). The apparatus was calibrated by photometrically measuring the actual orifice area. This orifice area was then used to determine the vapor pressure of a National Bureau of Standards sample of naphthalene over a pressure range of 0.1 to 1 torr.

The experimental vapor pressure of naphthalene thus obtained agreed with results reported in the literature⁷ to within $\pm 2\%$. The vapor pressure was calculated from the following equation:

$$P = \frac{17,134.5 \times \Delta W}{A \times t} \left(\frac{T}{MW} \right)^{\frac{1}{2}} \quad (1)$$

where

P = the vapor pressure in torr

ΔW = the weight loss in grams

t = the time for the recorded weight loss in seconds

A = the orifice area in cm^2

T = the absolute temperature in degrees Kelvin

MW = the molecular weight of a sample in grams/g mole

This method was used to measure vapor pressure in the range from 10^{-3} to 1 torr.

A DuPont differential thermal analyzer (DTA) model 900 was used previously to measure the upper vapor pressure range by the modified DTA method.^{3,8} The accuracy reported for this method was $\pm 5\%$.

The experimental vapor pressure data for each compound measured in this study were combined with vapor pressure data obtained by Belkin and Brown,³⁻⁵ and these data were then subjected to a computer analysis to derive an Antoine vapor pressure equation of the form:

$$\log P = A - B/(C+t) \quad (2)$$

where

P = vapor pressure in torr

t = temperature in degrees centigrade

A, B, C = constants

The standard deviation, SD, was calculated for each Antoine equation using the following equation:

$$SD = \left(\frac{S}{n-1} \right)^{\frac{1}{2}} \quad (3)$$

where

$$S = \sum (\log P_{\text{calc}} - \log P_{\text{exp}})^2$$

The logarithmic values were used since it has been reported⁹ that this procedure prevents excess weighting of the higher vapor pressure data.

The constants derived for the Antoine equation were used to calculate the heat of vaporization in kcal/mole and the volatility in g/m³ at any temperature from the following respective equations:

$$\Delta H_{\text{vaporization}} = 2.303 RB \left(\frac{T}{C+t} \right)^2 \quad (4)$$

$$\text{Volatility} = \frac{PM}{760 R'T} \quad (5)$$

where

B and C = Antoine equation constants

t = temperature in degrees centigrade

T = temperature in degrees kelvin

P = vapor pressure in torr

M = molecular weight in g/g mole

R = gas constant = 1.987×10^{-3} kcal/g mole °K

R' = gas constant = $82.05 \times 10^{-6} \frac{\text{atm-m}^3}{\text{g mole } ^\circ\text{K}}$

III. RESULTS.

The chemical purities and methods of analyses for the compounds studied in this work as well as that reported by Belkin and Brown^{3,5} are listed in table 1 together with the experimental methods used to measure the vapor pressure of each compound and the experimental temperatures covered.

The information given in table 2 provides a comparison between the experimental vapor pressure data (reported herein) with the vapor pressure calculated for the same temperature using the respective Antoine equations given in table 3. The Antoine equations used (table 3) represent those obtained from a consolidation of data provided in this report and also those data generated by Belkin and Brown.³⁻⁵ These comparative results are also shown graphically in the figure where our experimental data and the overall Antoine equation curves are shown. It is apparent that the calculated values provide a reasonably good fit to the experimental data. There exists some discrepancy in that data provided for EA 2223. This discrepancy results from the large number of experimental values generated by other investigators³ in their measurements over the temperature range from 60° to 225°C which influence the slope of the line.

Table 1. Chemical Purity and Method of Analysis of Chemical Agents Used

Compound	Vapor pressure method	Experimental range	Chemical purity	Method of analysis*
GD	Knudsen	-23 to 30	94 wt %	WM
			98.9 area %	GC
	Isoten/scope DTA	40 to 95 68 to 190	94 wt % 92.4 wt % 97 mole %	WM WM NMR
VX	Knudsen	30 to 40	91.9 wt %	WM
	Knudsen	52 to 100	89 wt %	WM
	DTA	110 to 231	91.7 wt %	WM
EA 2223	Knudsen	-1 to 50	94.0 wt %	WM
			99.6 area %	GC
	DTA DTA	60 to 221 60 to 221	94.0 wt % 87.6 wt %	WM WM
EA 3547	Knudsen	75 to 92	95 area %	GC
	DTA	182 to 335	95 area %	GC
EA 3580	Knudsen	58 to 90	99.4 wt %	WM
	DTA	208 to 286	99.7 wt %	WM
EA 5365	Knudsen	30 to 47 60 to 146	93 wt %	WM
			98 wt %	AH
			88 wt %	BH
EA 5533	Knudsen	18 to 44	97.9 area %	GC
	DTA	77 to 167	97.9 area %	GC

* WM = wet method
 GC = gas chromatography
 NMR = nuclear magnetic resonance spectroscopy
 AH = acid hydrolysis
 BH = base hydrolysis

Table 2. Experimental Versus Calculated Vapor Pressures of Chemical Agents

Temperature	Pressure (torr)		Percent difference $\frac{(\text{calc-exp})}{\text{exp}} (100)$
	Experimental	Calculated	
	(rounded values)		
°C	Compound GD		
-23.4	0.0053	0.0043	-16.1
-21.1	0.0053	0.0056	5.3
-20.0	0.0061	0.0064	4.6
-19.4	0.0072	0.0068	-4.6
-18.8	0.0068	0.0073	6.9
-16.6	0.0084	0.0093	10.7
-16.4	0.0095	0.0095	-0.4
-14.7	0.0113	0.0114	1.4
-13.6	0.0132	0.0128	-3.0
-12.8	0.0162	0.0140	-13.6
-11.1	0.0142	0.0167	17.5
-10.2	0.0178	0.0183	3.0
-9.8	0.0182	0.0191	4.8
-8.1	0.0213	0.0227	6.2
-1.7	0.0423	0.0423	-0.1
10.2	0.1344	0.1230	-8.5
17.0	0.2336	0.2156	-7.7
20.4	0.3255	0.2820	-13.4
25.0	0.4000	0.4006	0.2
30.0	0.6608	0.5782	-12.5
35.0	0.7000	0.8224	17.5
39.9	1.00	1.15	14.6
40.0	1.00	1.15	15.3
45.0	1.40	1.60	14.2
50.0	2.10	2.19	4.1
60.0	4.20	4.00	-5.8
64.8	5.20	5.18	-0.4
65.1	4.90	5.27	7.5
67.2	5.20	5.91	13.6
69.1	5.70	6.55	14.8
70.0	7.10	6.87	-3.2
71.2	6.90	7.32	6.1
73.9	7.60	8.43	10.9
74.0	8.40	8.47	0.9
76.7	9.80	9.73	-0.7
77.8	10.60	10.29	-2.9
80.1	12.00	11.55	-3.8
82.9	13.80	13.25	-3.9
85.2	15.60	14.81	-5.0
85.3	15.80	14.89	-5.8
85.6	16.00	15.10	-5.6

Table 2. (contd)

Temperature	Pressure (torr)		Percent difference $\frac{(\text{calc}-\text{exp})}{\text{exp}} (100)$
	Experimental	Calculated	
	(rounded values)		
°C			
86.5	16.10	15.76	-2.1
88.1	17.90	17.00	-5.0
90.0	18.80	18.59	-1.1
91.4	20.00	19.83	-0.8
92.1	21.60	20.48	-5.2
92.2	20.80	20.58	-1.1
93.9	23.50	22.24	-5.4
94.3	23.80	22.64	-4.9
95.1	22.50	23.47	4.3
Compound VX			
30.0	0.0013	0.0011	-11.9
35.0	0.0019	0.0020	2.8
40.0	0.0033	0.0034	3.7
52.0	0.0118	0.0113	-4.0
56.0	0.0172	0.0164	-4.7
57.3	0.0180	0.0184	2.3
59.6	0.0234	0.0226	-3.3
62.4	0.0276	0.0288	4.3
70.2	0.0505	0.0555	9.9
74.5	0.0659	0.0782	18.5
80.0	0.0995	0.1190	19.7
85.7	0.1937	0.1806	-6.7
88.5	0.2042	0.2202	7.8
90.5	0.2606	0.2531	-2.9
100.2	0.4684	0.4824	3.0
Compound EA 2223			
-0.7	0.0126	0.0112	-11.1
9.3	0.0304	0.0295	-2.8
19.6	0.0715	0.0738	3.2
30.2	0.1550	0.1746	12.6
40.0	0.3120	0.3624	16.2
50.0	0.6160	0.7215	17.1
Compound EA 3547			
74.9	0.0277	0.0268	-3.1
79.5	0.0377	0.0378	0.3
85.0	0.0559	0.0562	0.6
92.5	0.0911	0.0945	3.7

Table 2. (contd)

Temperature	Pressure (torr)		Percent difference $\frac{(\text{calc-exp})}{\text{exp}} (100)$
	Experimental	Calculated	
	(rounded values;		
°C	Compound EA 3580		
57.5	0.0004	0.0004	-7.0
68.0	0.0011	0.0011	-2.9
73.0	0.0017	0.0018	4.8
78.2	0.0028	0.0029	2.3
83.0	0.0042	0.0044	5.0
89.7	0.0077	0.0079	3.3
	Compound EA 5365		
29.9	0.0772	0.0734	-4.9
38.9	0.1465	0.1474	0.6
42.5	0.1874	0.1924	2.7
46.6	0.2478	0.2588	4.4
	Compound FA 5533		
18.0	0.0720	0.0729	1.3
20.0	0.0880	0.0856	-2.7
22.6	0.1020	0.1050	2.9
24.7	0.1240	0.1235	-0.4
27.0	0.1500	0.1471	-1.9
28.7	0.1660	0.1670	0.6
32.8	0.2250	0.2256	0.3
43.8	0.4900	0.4841	-1.2

Table 3. Combined Vapor Pressure Data

Agent	Antoine constants			Experimental range	Boiling point*	Standard deviation X 10 ⁻²
	A	B	C			
				°C	°C	log (torr)
GD	7.4706	1903.1	216.87	-23 to 190	198	3.4
VX	7.2810	2072.1	172.54	30 to 231	298	3.4
EA 2223	7.3092	1931.0	209.17	-1 to 221	227	3.0
EA 3547	7.6663	2579.7	204.32	75 to 335	335	0.9
EA 3580	7.5356	2523.2	172.14	58 to 286	370	1.8
EA 5365	8.6872	2778.9	253.04	30 to 146	226	1.6
EA 5533	8.0055	2387.6	243.16	18 to 167	223	1.2

*The temperature calculated from the Antoine equation at P = 760 torr.

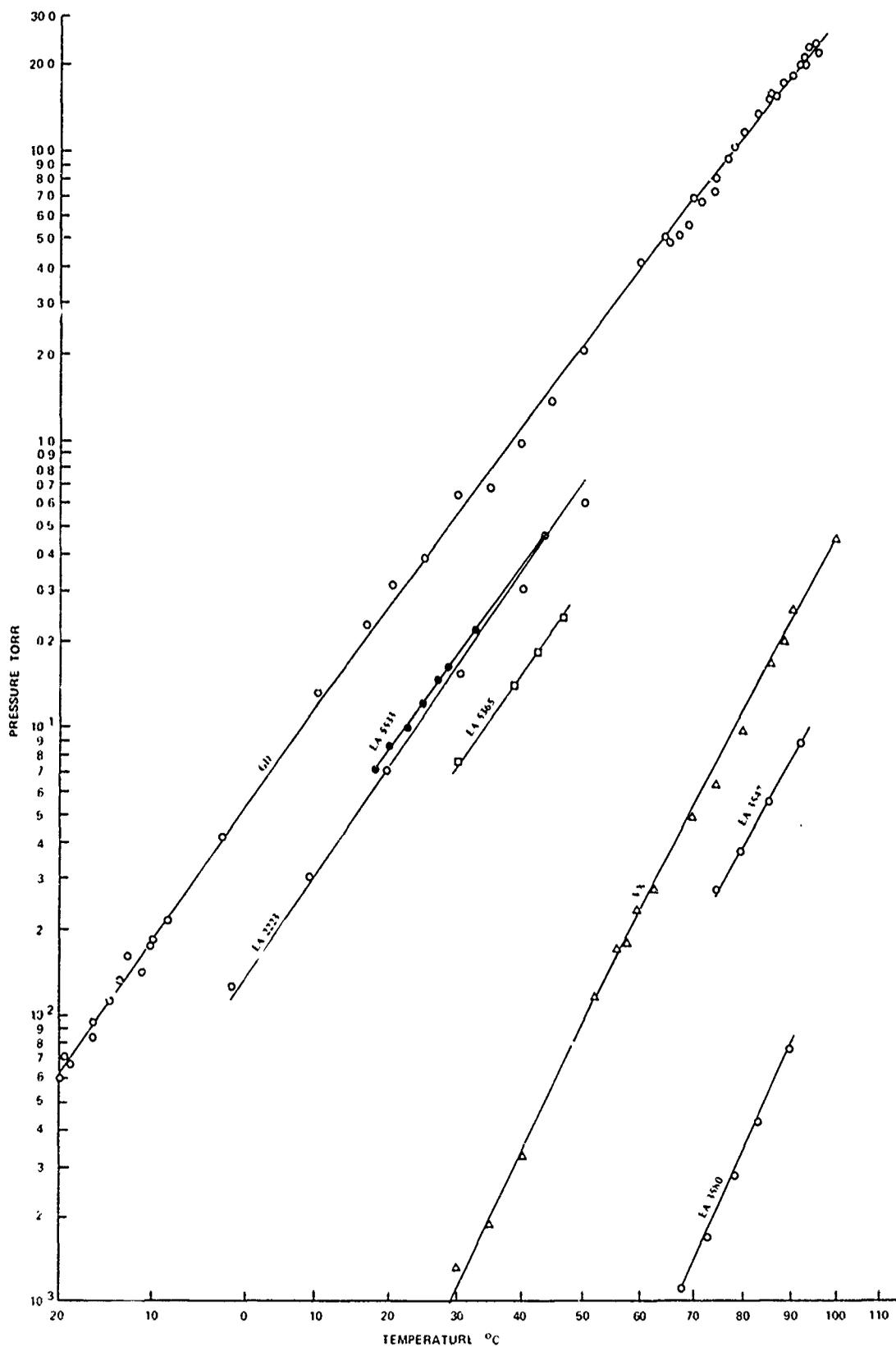


Figure. Experimental Vapor Pressure Points Versus Antoine Equation Lines for Compounds Given in Table 2

Table 3 provides the calculated Antoine vapor pressure equation constants and the experimental temperature range over which the combined experimental data for each compound were obtained. The normal boiling point, which was calculated from the Antoine vapor pressure equation assuming a vapor pressure of 760 torr, as well as the standard deviation of the experimental data calculated using equation 3, are given in table 3.

Table 4 provides ready access to calculated values of vapor pressure, volatility, and heat of vaporization at selected temperatures using the Antoine equations in table 3 for the chemical agents studied.

IV. DISCUSSION.

As a result of this work, vapor pressure data in the range 0.001 to 1 torr are available for samples of candidate chemical agents EA 2223, EA 3547, EA 5365, and EA 5533 of known purity. In addition, an extensive vapor pressure study is also reported for chemical agents GD and VX. As can be seen in the figure, this range covers vapor pressures which would occur at environmental temperatures for most of these chemical agents.

The vapor pressure data for GD reported here is the most extensive study reported to date and incorporates two different experimental measurement methods. All of the available literature references found have been limited to either one experimental method^{5,10,11} or to measurements over small temperature ranges. Our results agree to within $\pm 3.7\%$ of the data obtained by DTA⁵ and reported between 2 torr and 30 torr. The vapor pressure of GD at 25°C measured in this work was found to be 0.40 torr, compared to previously reported values of 0.31 torr¹⁰ and 0.51 torr¹¹ at this temperature. The discrepancy in these results could be due to differences in the purity of the GD samples used; unfortunately, neither literature reference^{10,11} reported the purity of the GD used. Zeffert and Couiter¹⁰ indicated that decomposition of the sample occurred during their measurements, and this could explain their lower value at 25°C when compared with our results. The agent used in the current work was redistilled from a Teflon spinning-band annular still at reduced pressure and was analyzed before use.

The vapor pressure of chemical agent VX measured at 30°C was found to be 1.3×10^{-3} torr, which is in good agreement with the previously reported¹³ value of 1.5×10^{-3} torr at this temperature.

The only literature references to the vapor pressure of candidate chemical agents EA 2223, EA 3547, EA 5365, and EA 5533 were those published by Belkin and Brown^{3,4} using the DTA method. They report the vapor pressure of these compounds at higher temperatures than are reported herein. A 10°C extrapolation of the DTA vapor pressure data provides estimated vapor pressure values at 50°C for EA 2223 and EA 5365 of 0.96 and 0.35 torr, respectively, versus our respective experimental values at this temperature of 0.72 and 0.33 torr. The agreement between the two methods is good for EA 5365 at this temperature, whereas the poorer agreement for the vapor pressure of EA 2223 may be due to the lower purity of a sample used in the DTA work or to the fact that the accuracy of the DTA method is reported to be poor at pressures of approximately 1 torr. The DTA method has been shown³ to be less accurate at 1 torr since the ability to control and measure pressure in this region was limited.

Table 4. Properties of Chemical Agents at Selected Temperatures

Compound	Temperature	Pressure	Volatility	Heat of vaporization
	°C	torr	gm/cu m	kcal/mole
GD	-25*	0.0036	0.042	14.57
	0	0.0496	0.531	13.82
	25	0.401	3.93	13.23
	50	2.19	19.8	12.77
	100	29.17	228.4	12.08
	150	192.0	1325.9	11.59
VX	25*	0.0006	0.009	21.60
	50	0.0093	0.123	20.00
	100	0.477	5.48	17.78
	150	7.19	72.9	16.32
	200	52.35	474.4	15.30
	250*	238.3	1953.0	14.54
EA 2223	0	0.0120	0.138	15.07
	25	0.116	1.22	14.33
	50	0.722	7.03	13.74
	100	11.57	97.6	12.87
	150	85.69	637.1	12.27
	200	389.0	2586.0	11.82
EA 3547	75	0.0270	0.243	18.34
	100	0.155	1.30	17.75
	150	2.43	18.0	16.84
	200	19.32	127.8	16.17
	250	97.32	582.3	15.65
	300	355.7	1943.0	15.25
EA 3580	75	0.0021	0.029	22.92
	100	0.018	0.235	21.71
	150	0.51	5.81	19.92
	200	5.70	58.6	18.67
	250	36.2	337.0	17.73
	300*	155.5	1320.0	17.02
	350*	505.1	3943.0	16.45
EA 5365	25*	0.049	0.53	14.62
	50	0.33	3.23	14.46
	100	6.54	55.7	14.21
	150*	62.0	466.0	14.02
	200*	357.5	2401.0	13.87
EA 5533	25	0.0571	0.535	13.61
	50	0.7261	6.060	13.28
	100	11.16	80.7	12.92
	150	85.62	545.7	12.66
	200*	414.7	2364.1	12.46

*Extrapolated point

The vapor pressure of the candidate chemical agent EA 3547 measured at 90°C in this study agreed within 7.6% with the DTA results extrapolated to this temperature. For candidate chemical agent EA 5533, the vapor pressure measured at 50°C was 0.73 torr, and the value obtained by extrapolation of the DTA data was also 0.73 torr.

Two sets of vapor pressure data have been reported for candidate chemical agents EA 3580.^{5,12} A vapor pressure of 8.1×10^{-3} torr was measured at 90°C in this study, compared to an extrapolated value from DTA data of 7.9×10^{-3} torr,⁵ and an experimental value of 11.7×10^{-3} torr reported by Benning¹² using the Knudsen method.

Most of the new data reported herein were obtained by the Knudsen effusion technique, which is limited to a pressure range from 10^{-3} torr to approximately 1 torr. The previously reported data,³⁻⁵ obtained by DTA, were limited to pressures above 1 torr and to temperatures above 35°C. Consequently, extrapolation of DTA data or of Knudsen effusion data alone to obtain the vapor pressure of chemical agents outside of the measured range can be quite inaccurate when a long extrapolation is required. The extrapolated vapor pressure of VX at 30°C from DTA data³ measured from 110° to 231°C is 5.5×10^{-3} torr, whereas the experimentally measured value reported herein is 1.3×10^{-3} torr. Similar errors would occur if the Knudsen data were extrapolated into the upper pressure range of the DTA results. However, when the Knudsen vapor pressure data for VX and the previously reported DTA data were combined, the calculated vapor pressure (using the equation in table 3) was 1.1×10^{-3} torr. This estimate agrees well with the actual experimental value given above.

The validity of combining Knudsen, isoteniscope, and DTA vapor pressure results is demonstrated in the figure. The experimental data obtained in our study by the Knudsen and isoteniscope methods, which are represented as points in the figure, fit the combined-data Antoine equation curves very well except for EA 2223. The poor fit of the Knudsen vapor pressure values to the combined-data Antoine curve for EA 2223 may be due to the difference in the purity of the two samples used by the DTA and Knudsen methods or to the difference in the number of experimental points measured by each method. The number of experimental vapor pressure points reported for EA 2223 by the DTA method³ was far greater than the number of points measured by the Knudsen method provided in this report, and several of the DTA points were in a pressure region where the DTA method is known to be less accurate. Consequently, since the Antoine curve generated from the combined data emphasizes the DTA results, the inclusion of DTA data in the region of 1 torr may account for the discrepancy in the EA 2223 results shown in the figure.

V. CONCLUSIONS.

1. Data are reported for vapor pressures between 10^{-3} torr and 1 torr for candidate chemical agents EA 2223, EA 3547, EA 5365, and EA 5533 as measured by the Knudsen technique. Vapor pressure measurements are also reported for the candidate chemical agent EA 3580 and for chemical agents GD and VX.

2. Vapor pressure data for chemical agents obtained by the Knudsen effusion technique and the isoteniscope method were consolidated with data obtained at higher temperatures by the modified differential thermal analysis (DTA) method, and Antoine vapor pressure equations were derived for predictive use. Predictions of vapor pressures made from these Antoine equations appear to be superior to estimates made by extrapolation of data over a narrower data range.

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MEMORANDUM FOR Defense Technical Information Center (DTIC), 8725 John J. Kingman Road, Fort Belvoir, VA 22060-6218.

SUBJECT: Change in Limitation (Public Release of Information)

1. The purpose of this memorandum is to recommend the Release of Information to the General Public regarding Edgewood Arsenal Technical Memorandum (EATM 134-2), Viscosity of VX in the Temperature Range -4 degrees - -40 degrees F, dated May 1969; and Edgewood Arsenal Technical Report (EC-TR-76058), The Vapor Pressure of Chemical Agents GD, VX, EA 2223, EA 3547, EA 3580, EA 5365 and EA 5593, dated August 1976. The first memorandum was Confidential and was previously downgraded to Unclassified on 20 Mar 1978. The DTIC Number is AD-501931. The second report is Unclassified and the DTIC Number is ADB-013164.
2. This document has been reviewed by Subject Matter Experts from the Edgewood Chemical Biological Center (ECBC) on Aberdeen Proving Ground, Maryland and deemed releasable to the General Public. Request that this document be properly identified and appropriately marked.
3. As the Security Manager for the documents in question, I concur with the recommendations made by the ECBC Review Team.
4. Point of contact for this action is the Information Security Officer, Ronald Stafford at 410-436-6810 or the undersigned at 410-436-7232.

Encl

June K. Sellers

JUNE K. SELLERS
ECBC Security and Surety Manager