VAPOR PRESSURE OF *N,N'*-DIISOPROPYLCARBODIIMIDE (DICDI)

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14. ABSTRACT:

N,N′-diisopropylcarbodiimide, also known as DICDI, is a stabilizer for chemical warfare nerve agents such as VX and GD. The vapor pressure of DICDI was measured using complementary methods that allow data collection at ambient and high temperatures using gas saturation and differential scanning calorimetry, respectively. A three-parameter Antoine correlation equation based on the new data was determined. The resulting calculated values compared favorably to published and unpublished data cited in this report. This report includes derived properties, such as volatility, temperature-dependent heats of vaporization, normal boiling point, and entropy of vaporization.

15. SUBJECT TERMS

N,N′-Diisopropylcarbodiimide (DICDI)	Vapor pressure	Volatility	Enthalpy of vaporization
Entropy of vaporization	Antoine correlation equation	Gas saturation	Normal boiling point
Differential scanning calorimetry (DSC)			

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PREFACE

The work described in this report was authorized under the Chemical and Biological Defense Technology base program. The work was started in April 2009 and completed in December 2011.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

Differential scanning calorimetry data reported herein are documented in the U.S. Army Edgewood Chemical Biological Center (ECBC) notebook number 06-0114. Saturator data are documented in ECBC notebook number 05-140.

This report has been approved for public release.

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VAPOR PRESSURE OF *N,N′*-DIISOPROPYLCARBODIIMIDE (DICDI)

1.   INTRODUCTION

Vapor pressure is a physical property that is critical for understanding the behavior of chemicals in the laboratory as well as in the environment. Knowledge of vapor pressure is also important for a number of applications related to chemical warfare defense, including the generation of precisely controlled challenge concentrations for quantitative toxicology evaluations and detector testing as well as the prediction of chemical removal efficiency by air filtration systems. Vapor pressure data can also be useful for estimating the concentration of solution mixtures by quantitative analysis of head space. This type of analysis can be performed if the vapor pressures of the major components in a mixture are known, which is the reason for our interest in *N,N′*-diisopropylcarbodiimide (DICDI; Chemical Abstracts Service [CAS] no. 693-13-0). DICDI has been used as a stabilizer for chemical warfare agents,[1] and could be considered a signature compound for those materials. This report documents new vapor pressure data measured in our laboratory for DICDI (Figure 1) and a correlation based on those data.
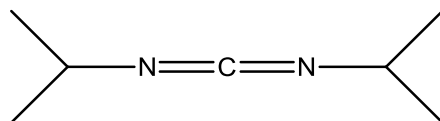
Figure 1. Structure of DICDI.

Patents and literature reports contain DICDI vapor pressure data.[2–14] In 1962, the American Cyanamid Corporation (now Cytec Industries; Woodland Park, NJ), under contract to the U.S. Army, reported smoothed vapor pressure values based on isoteniscope measurements obtained between 50 and 125 °C.[15] That report is shown in the appendix. These historical data are compared to the new correlation.

2.   EXPERIMENTAL DETAILS

The DICDI used in the present work was obtained from Sigma-Aldrich (St. Louis, MO), catalog number D125407, 99%, and was used as received. Two ASTM International (West Conshohocken, PA) methods[16,17] were modified for use in measuring the vapor pressure of DICDI. The first ASTM method involves gas saturation. In the current work, the mass loss of DICDI, as a result of purging with an inert gas carrier at a known temperature and rate, was measured. The second ASTM method employs the differential scanning calorimeter (DSC) pinhole technique. Recent work in our laboratory has extended the useful range for DSC vapor pressure data measurement to below 200 Pa.[18]

2.1 Vapor Pressure by Gas Saturation

Before gas saturation data were collected, the DICDI was purged in the saturator cell using dry nitrogen (dew-point temperature of <200 K) at a flow rate of 250 standard cm^3/min (sccm) and at ambient conditions of temperature and pressure to remove volatile impurities. After the purge step, two separate trials were run to measure the vapor pressure. Procedures were similar, with differences only as noted below. The saturator cell was weighed on a Sartorius (Bohemia, NY) BP211D analytical balance, with a repeatability of ≤0.2 mg and a deviation from linearity of ≤0.1 mg within the range of masses in our study. The temperature of the DICDI was held at 15.0 °C by submerging the saturator cell into a Julabo (Allentown, PA) F25 constant-temperature bath. The temperature of the bath was measured using a calibrated Miller and Weber (New York, NY) T-7755 thermometer, with a stated accuracy of ±0.05 K. The saturator cell temperature was then equilibrated for ≥10 min before the carrier gas flow was started. The carrier gas flow was initiated, and the flow rate was carefully measured using a Brooks (Hatfield, PA) model 5850S mass flow controller for a pre-determined time. The saturator cell was then removed from the bath, the external surfaces were dried with a lint-free paper towel, and the saturator cell was set aside for >2 h to let it re-equilibrate to ambient temperature and permit any remaining trace water on the external surfaces to evaporate. The saturator cell mass was then remeasured to determine mass loss. The only difference in the two trials was that one was run using a carrier gas flow rate of 99.9 sccm, and the second was run at 35.0 sccm to demonstrate that the resulting calculated vapor pressure did not depend on flow rate and, thus, that the saturator output contained saturated vapor.

Ambient pressure was measured continuously during each run using an Omega (Stamford, CT) DPI 740 digital pressure calibrator (stated accuracy, 0.02%). These data were used in the vapor pressure calculation as described herein. The pressure difference between the sampling location and ambient was determined to be less than 10 Pa (<0.01% of ambient pressure) at the flow rate used in this work; therefore, no corrections were made. The bath temperature, ambient pressure, and carrier gas flow rate were recorded at 4 s intervals using a Labview (National Instruments; Stamford, CT) program. The vapor pressure was calculated using

$$P = P_{amb} \cdot n_a/(n_{car} + n_a) \tag{1}$$

where P is vapor pressure of analyte; P_{amb} is ambient atmospheric pressure, which is the sum of the partial pressures of the DICDI ($P_{analyte}$) and the nitrogen carrier gas ($P_{carrier}$); n_a is moles of analyte, determined gravimetrically; and n_{car} is moles of carrier gas, determined by multiplying the carrier gas mass flow rate by the carrier gas flow time.

2.2 Vapor Pressure by Differential Scanning Calorimetry

Vapor pressures at higher temperatures were measured using a TA Instruments, Inc. (Newcastle, DE) 910 DSC with a 2200 controller. The system vacuum was achieved using a rotary vane pump with coarse and fine pressure regulators to hold the vacuum constant to ±0.01 kPa as the specimen boiled. Cell pressure was measured using a mercury manometer that had been calibrated using the vapor pressure of water at several temperatures over the operational

range. Small specimens (~4 μL) of the test material were contained in TA Instruments hermetically sealed sample pans, each with a single 75 μm pinhole in the lid. The measurement consisted of heating the specimen in the DSC cell through the boiling temperature at a controlled rate (5 K/min), while the pressure in the cell was held constant. At the boiling temperature, the vaporized specimen escaped from the sample pan through the pinhole in the lid. The energy associated with the transition from liquid to vapor was recorded as a sharp boiling endotherm. The boiling point was taken at the intersection of tangents to the heat flow versus temperature curve at the onset of boiling. The experimental pressure was the pressure in the cell as the specimen boiled. This process was repeated with new specimens at different pressures to obtain a series of points that were used as input to the vapor pressure correlation curve.

Measurements with DICDI were completed between 55.46 and 148.27 °C (3,490 and 101,530 Pa).

Before the DICDI measurements were made, the DSC was calibrated using indium in accordance with ASTM Practice E967-08, *Standard Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers.*[19] The observed melting onset temperature was within 0.2 K of the literature value.

Data generated by the saturator and DSC methods were combined and fitted to the Antoine equation[20] to generate the vapor pressure curve using

$$\ln(P) = a - b/(c + T) \tag{2}$$

where P is vapor pressure (Pa); a, b, and c are Antoine equation coefficients; and T is temperature (K).

The Antoine coefficients were derived by minimizing the sum of the squares of the natural logarithms of differences between measured and calculated values.

Enthalpy of vaporization was calculated as a function of temperature using

$$\Delta H_{vap} = b \cdot R \cdot T^2/(c + T)^2 \tag{3}$$

where ΔH_{vap} is enthalpy of vaporization and R is the gas constant, 8.3144 J/(K·mol).

Saturation concentration, C_{sat}, also referred to as volatility, is defined as the concentration of the saturated vapor in units of milligrams per cubic meter at a given temperature and is used extensively by the toxicology community, especially in reference to inhalation hazard testing. Volatility is calculated using

$$C_{sat} = PM/RT \tag{4}$$

where C_{sat} is saturation concentration (mg/m^3), and M is molecular mass (g/mol).

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The entropy of vaporization was calculated by dividing the enthalpy of vaporization at the normal boiling point by the normal boiling point temperature

$$\Delta S_{vap} = \Delta H_{vap,NBPt}/T_{NBPt} \tag{5}$$

where ΔS_{vap} is entropy of vaporization (J/mol-K), $\Delta H_{vap,NBPt}$ is enthalpy of vaporization at the normal boiling point (J/mol), and T_{NBPt} is normal boiling point temperature (K).

3.   RESULTS

Table 1 lists the vapor pressure data for DICDI that were determined in the present work using the saturator and DSC methods, along with the Antoine equation. Table 1 also includes the calculated vapor pressure values based on the Antoine equation and the percent differences between experimental and calculated values at each experimental temperature. The gas saturation and DSC data and resulting correlations are shown in Figure 2. The two saturator experiments, run at 35.0 and 99.9 sccm, resulted in values of 324.5 and 322.0 Pa or 2.434 and 2.415 Torr, respectively, which demonstrates that the system was at or near equilibrium at both flow rates. The average value, rounded to the nearest 1 Pa, was used for the correlation calculation.

Table 2 provides a list of values from the literature,[2–14] along with calculated values based on the new Antoine equation. Figure 3 illustrates the agreement between the literature data and the Antoine correlation equation presented herein.

Table 3 provides a list of values from the work performed by the American Cyanamid Corporation[15] while under contract to the U.S. Army and compares those values to values calculated using our new Antoine equation. Figure 3 compares these data to the Antoine correlation equation presented in this report.

Vapor pressure, saturation concentration (or volatility), and enthalpy of vaporization calculated from the Antoine coefficients for DICDI are listed at selected temperatures in Table 4.

Based on the data and correlation, the normal boiling point calculated for DICDI is 148.06 °C. The calculated entropy of vaporization is 93.37 J/mol-K, which is in good agreement with the value expected on the basis of Trouton's rule.

Table 1. New DICDI Vapor Pressure Data, Antoine Correlation
Equation, and Comparisons to Calculated Values

Temperature (°C)	Experimental Vapor Pressure (P_{expt})		Calculated Vapor Pressure (P_{calc})		Percent Difference[*]
	Pa	Torr	Pa	Torr	
Gas Saturation					
15.0	323	2.42	322.1	2.416	0.28
DSC					
55.46	3490	26.2	3497	26.23	–0.20
65.45	5500	41.3	5632	42.24	–2.34
77.06	9480	71.1	9384	70.38	1.02
87.90	14860	111.4	14560	109.2	2.06
111.15	33560	251.7	33690	252.7	–0.39
148.27	101530	761.6	101900	764.3	–0.36
$\ln(P_{Pa}) = 20.78393 - 3214.75/(T_K - 73.96220)$ $\log(P_{Torr}) = 6.901441 - 1396.15/(T_C + 199.1878)$					

[*] Percent difference = $100 \cdot (P_{expt} - P_{calc})/P_{calc}$.
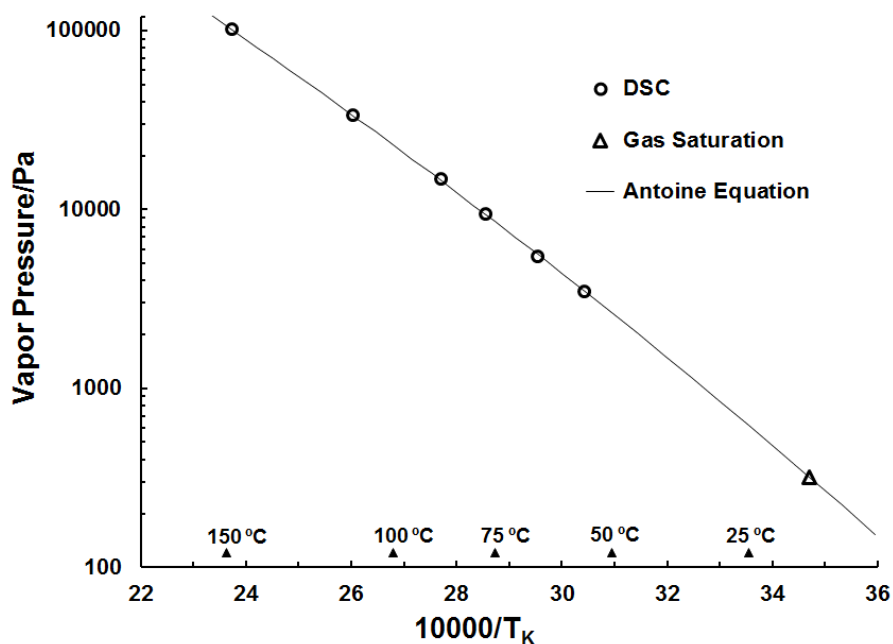T_K, temperature in Kelvin.
T_C, temperature in Celsius.

Figure 2. New vapor pressure data and Antoine correlation equation for DICDI.

Table 2. Literature Values for DICDI Vapor Pressure and Comparison
to Values Calculated Using the New Correlation

Temperature (°C)	Experimental Vapor Pressure (P_{expt})		Calculated Vapor Pressure (P_{calc})		Percent Difference[*]	Literature Reference
	Pa	Torr	Pa	Torr		
35.5	1200	9	1195	8.963	0.42	14
36.5	1333	10	1267	9.500	5.21	6
36.5	1333	10	1267	9.500	5.21	7
36.5	1333	10	1267	9.500	5.21	9,13
43.5	1867	14	1877	14.08	−0.53	10
44	2000	15	1929	14.47	3.68	12
45	2133	16	2036	15.27	4.76	8
53	3333	25	3092	23.19	7.79	5
84.5	12000	90	12730	95.50	−5.73	5
144.4	101325	760	91810	688.6	10.36	4
147	101325	760	98500	738.8	2.87	11
157.5	101325	760	129500	971.0	−21.76	3
160	101325	760	137800	1034	−26.47	2

[*]Percent difference = $100 \cdot (P_{expt} − P_{calc})/P_{calc}$.

Table 3. Unpublished DICDI Vapor Pressure Values from Literature Reference 15
and Comparison to Values Calculated Using the New Correlation

Temp (°C)	Experimental Vapor Pressure[a] (P_{expt})		Calculated Vapor Pressure (P_{calc})		Percent Difference[b]
	Torr	(Pa)	Torr	Pa	
51	20	2670	20.94	2792	−4.37
71	50	6670	54.21	7228	−7.72
87	100	13330	105.4	14060	−5.19
105	200	26660	205.0	27330	−2.45
125	400	53330	393.4	52450	1.68
146[c]	760	101325	719.2	95880	5.68

[a]Based on smoothed isoteniscope data.

[b]$100 \cdot (P_{expt} − P_{calc})/P_{calc}$.
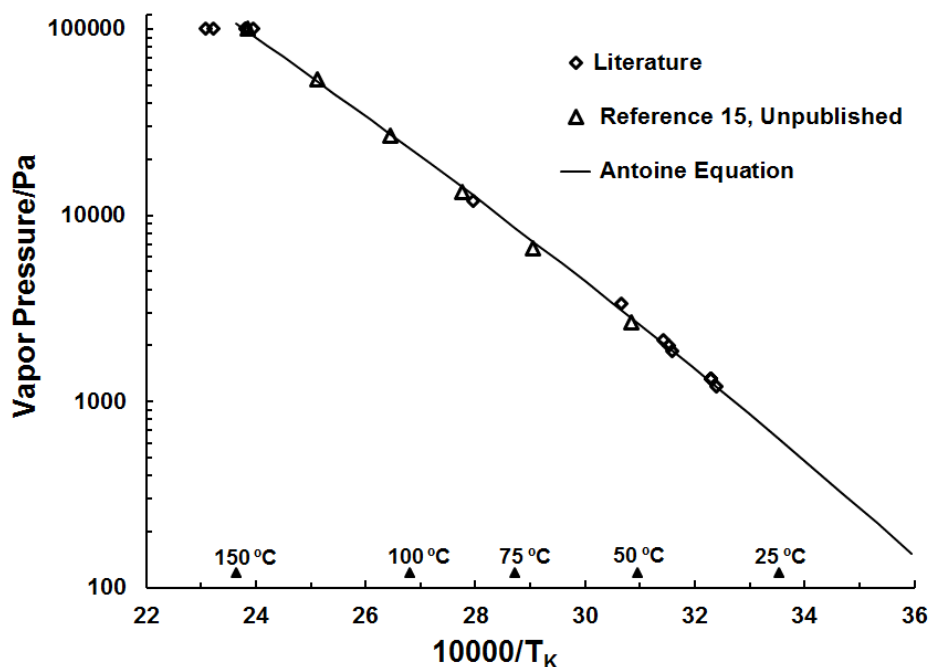
[c]Extrapolated.

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Figure 3. Literature and unpublished isoteniscope DICDI vapor
pressure data and Antoine correlation equation.

4.   DISCUSSION

Work from our laboratory has resulted in reports on the vapor pressure of
chemical warfare agents (CWAs) and their surrogates, precursors, and decomposition
products.[21–28] This report is concerned with DICDI, which has been used as a nerve agent
stabilizer, and like decomposition products and precursors, may be considered to be a signature
compound; that is, a chemical whose detection provides evidence of the presence of a CWA of
interest.

Data quality is assessed on the basis of scatter within each data set and agreement
among data measured using more than one analytical method. The internal agreement among the
seven new data points presented in this report is as good as any data produced in our ECBC
laboratory. However, the agreement between data that was measured using two different
methods provides stronger evidence that the data are reliable. Historically, work in our
laboratory has been based on a combination of methods, including differential thermal analysis
and Knudsen effusion.[25] The former has been replaced by a similar thermal analysis method,
DSC, and the latter has been replaced in our laboratory by methods that are based on gas
saturation because of its relative insensitivity to impurities when combined with analysis
involving separation of analytes, such as gas chromatography (GC). This advantage was
demonstrated most clearly in our work with VX (*O*-ethyl *S*-(2-diisopropylaminoethyl) methyl
phosphonothioate), which was found to be a minor headspace component, even at relatively high
liquid-phase mole fractions.[22] In the present case, the vapor pressure of DICDI at 25 °C is nearly

4 orders of magnitude greater than that of VX. According to Raoult's law, the partial pressure of DICDI at a 0.01 mole ratio in VX would be more than 50 times higher than that of VX in the headspace above the liquid. High-resolution, vapor-phase infrared spectra of the effluent from a saturator cell that was freshly filled with weapons-grade VX showed that the vapor was dominated by spectral features associated with DICDI, whereas the VX was not initially observed.[29] The earlier work from our laboratory successfully distinguished VX from a myriad of impurity peaks that were present in the head space above VX. As a result, we were able to obtain accurate vapor pressure data, despite the relatively meager amount of VX in the headspace above a >0.95 mole fraction of liquid VX.[22]

Table 4. Calculated Vapor Pressure, C_{sat}, and ΔH_{vap} for DICDI at Selected Temperatures

Temperature (°C)	Vapor Pressure		C_{sat} (mg/m^3)	ΔH_{vap} (kJ/mol)
	Torr	Pa		
−40	0.01352	1.803	117.4	57.34
−30	0.04460	5.947	371.2	55.21
−20	0.1288	17.17	1029	53.35
−10	0.3325	44.33	2557	51.71
0	0.7802	104.0	5779	50.26
5	1.158	154.4	8425	49.60
10	1.688	225.0	12060	48.97
15	2.416	322.1	16970	48.38
20	3.402	453.6	23490	47.81
25	4.719	629.2	32030	47.27
30	6.452	860.3	43070	46.76
40	11.60	1546	74930	45.82
50	19.89	2652	124600	44.95
60	32.72	4362	198700	44.16
70	51.87	6916	305900	43.43
80	79.56	10610	456000	42.77
90	118.5	15800	660400	42.15
100	171.8	22900	931500	41.58
120	336.8	44900	1733000	40.55
140	609.9	81320	2988000	39.66
148.06	760	101325	3651000	39.33

Several variations of the gas saturation method have been used in the literature, including mass loss (as described herein),[23] direct effluent injection into a GC system,[26] effluent concentration in combination with GC analysis,[22] and denuder collection in combination with liquid chromatography (LC) and mass spectrometry (MS).[27] Each of these methods has advantages depending on the analyte of interest. Mass loss is the simplest method, but it is only suitable for higher-volatility materials that are available in high-purity form. Direct-sampling in combination with GC analysis is more sensitive than the mass-loss method but not as sensitive as the vapor-concentration method, which has proven to be most useful for low-volatility, thermally stable analytes. The denuder–LC–MS method has proven to be the most sensitive and works well for materials that are thermally labile.

As seen in Table 1 and Figure 2, DICDI vapor pressure data, measured in our laboratory using DSC and vapor saturation, are among the most consistent data that we are aware of, as indicated by the small differences between experimental and correlated values. Further evidence of their validity is provided by comparisons to literature data[2–14] and to the data measured by American Cyanamid personnel while under contract to the U.S. Army.[15] These data were compared to our new correlation in Figure 3 and Tables 2 and 3. None of the prior data were used to determine the new correlation presented in this report. We found the distillation data to be erratic, and the American Cyanamid values were derived from measured data.

In the ambient temperature range, the vapor pressure of DICDI is about 1.7 times that of isopropyl methylphosphonofluoridate (GB),[28] which is the most volatile of the classical chemical warfare nerve agents. Comparison of GB and DICDI vapor pressure correlation equations over a broad range of temperatures is shown in Figure 4.
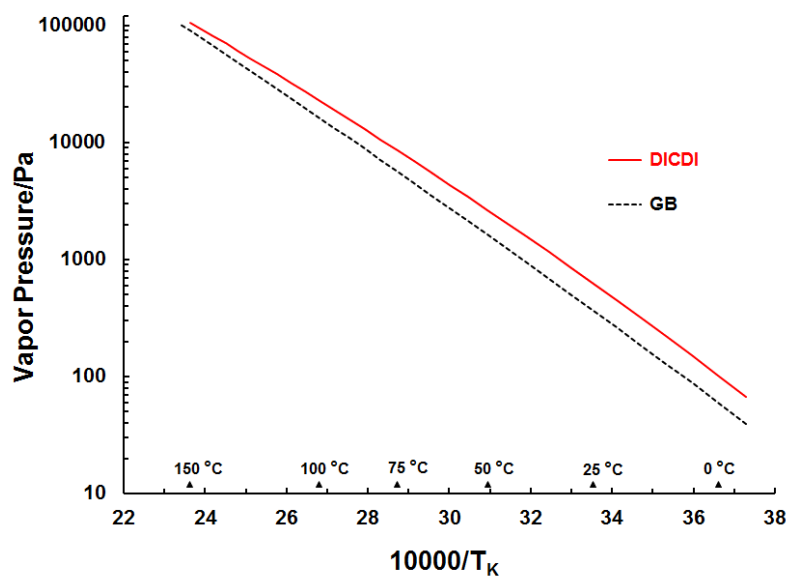

Figure 4. Comparison of DICDI vapor pressure to that of GB.

5.   CONCLUSIONS

New vapor pressure data for DICDI determined using DSC and gas saturation and reported herein are in good agreement, which resulted in a high degree of confidence in the accuracy of each experimental technique. The vapor pressure correlation developed using these data enables the interpolation and limited extrapolation of data over a large temperature range and is consistent with literature data and with the data contained in the unpublished American Cyanamid letter report.

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LITERATURE CITED

1.   Rohrbaugh, D.K.; Hondrogiannis, G.; Yang, Y.C. *Analytical Method and Detection Limit Studies for Detection of GB in GB Hydrolysate*; ECBC-TR-509; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2006; UNCLASSIFIED Report (ADA456409).

2.   Smeltz, K.C. Catalysts for Preparing Carbodiimides. U.S. Patent 3,157,662, November 17, 1964.

3.   Lecher, H.Z.; Parker, R.P.; Long, R.S. Preparation of 1,2-Disubstituted-3-Cyanoguanidines. U.S. Patent 2,479,498, September 1949.

4.   Ruby, W.R. Preparation of *N,N′*-Dicyclohexyl Carbodiimide. U.S. Patent 3,201,463, August 17, 1965.

5.   Budnick, E.G. Triarylarsines as Catalysts for Converting Isocyanates to Carbodiimides. U.S. Patent 3,406,198, October 15, 1968.

6.   Coles, R.F. Process of Producing Carbodiimides. U.S. Patent 2,946,819, July 26, 1960.

7.   Bayer Corporation, Carbodiimides. U.K. Patent GB685,970, January 14, 1953.

8.   Seefelder, M.; Neubauer, G. Verfahren zur Herstellung von Carbodiimiden. German Patent 1,125,914, October 1962.

9.   Schmidt, E.; Reichenhall, B.; Schnegg, R. Verfahren zur Herstellung von Carbodiimiden. German Patent 823,445, December 1951.

10.   Hinton, I.G.; Webb, R.F. The Disproportionation of Carbodiimides. *J. Chem. Soc.* **1961**, 5051–5052.

11.   Furumoto, S. The Synthesis of Carbodiimides from *N,N′*-Disubstituted Thioureas and 2-Chloro-4,6-dimethylpyrimidine, 2,4-Dichloropyrimidine or *N*-Phenylbenzimidoyl Chloride. *Journal of Synthetic Organic Chemistry, Japan* **1975**, *33*, 748–752.

12.   Kagami, H.; Hanzawa, N.; Suzuki, N.; Yamaguchi, S.; Saito, M.; Motoki, S. Nucleophilic Substitution on Dialkoxy Disulfides. III. Reaction with Thioureas. *Bull. Chem. Soc. Jpn.* **1980**, *53*, 3658–3660.

13.   Schmidt, E.; Striewsky, W. Zur Kenntnis aliphatisher Carbodiimide (III. Mittel). *Berichte der Deutschen Chemischen Gesellschaft Abteilung B: Abhandlungen* **1941**, *74B*, 1285–1296.

14.   Eilingsfeld, H.; Neubauer, G.; Seefelder, M.; Weidincer, H. Synthesen mit Amaidchloriden, III. Synthese und Reaktionen von Chlorformamidiniumchloriden. *Chemische Berichte* **1964**, *97*(5), 1232–1245.

15.   Groth, E., Jr. American Cyanamid Corporation, Bound Brook, NJ. Letter report to the U.S. Army, 1962. (See Appendix.)

16.   ASTM International. *Standard Test Method for Determining Vapor Pressure by Thermal Analysis*; ASTM E 1782; West Conshohocken, PA, 2014.

17.   ASTM International. *Standard Test Method for Vapor Pressure*; ASTM E 1194; West Conshohocken, PA, 2001.

18.   Brozena, A. Vapor Pressure of 1-Octanol below 5 kPa using DSC. *Thermochim. Acta* **2013**, *561*, 72−76.

19.   ASTM International. *Standard Test Method for Temperature Calibration of Differential Scanning Calorimeters and Differential Thermal Analyzers*, ASTM E 967; West Conshohocken, PA, 2008.

20.   Thomson, G.W. The Antoine Equation for Vapor-Pressure Data. *Chem. Rev*. **1946**, *38*, 1−39.

21.   Abercrombie-Thomas, P.L.; Butrow, A.B.; Buchanan, J.H. *Selected Physical Properties of 2-Chloroethyl-3-chloropropyl Sulfide (CECPRS)*; ECBC-TR-804; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2010; UNCLASSIFIED Report (ADA531948).

22.   Tevault, D.E.; Brozena, A.; Buchanan, J.H.; Abercrombie-Thomas, P.L.; Buettner, L.C. Thermophysical Properties of VX and RVX. *J. Chem. Eng. Data* **2012**, *57*, 1970−1977.

23.   Butrow, A.B.; Buchanan, J.H.; Tevault, D.E. Vapor Pressure of Organophosphorus Nerve Agent Simulant Compounds. *J. Chem. Eng. Data* **2009**, *54*, 1876–1883.

24.   Williams, B.R.; Hulet, M.S.; Brozena, A.; Miles, Jr., R.W.; Tevault, D.E. Vapor Pressure of 2-Dialkyl Aminoethanethiols. *J. Chem. Eng. Data* **2013**, *58*, 1679–1684.

25.   Brozena, A.; Buchanan, J.H.; Miles, R.W., Jr.; Williams, B.R.; Hulet, M.S. Vapor Pressure of Triethyl and Tri-*n*-Propyl Phosphates and Diethyl Malonate. *J. Chem. Eng. Data* **2014**, *59*, 2649–2659.

26.   Tevault, D.E.; Buchanan, J.H.; Buettner, L.C.; Matson, K.L. *Vapor Pressure of Cyclohexyl Methylphosphonofluoridate (GF)*; ECBC-TR-304; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2009; UNCLASSIFIED Report (ADA503835).

27.   Jenkins, A.L.; Bruni, E.J. Buettner, L.C.; Sohrabi, A.; Ellzy, M.W. *Vapor Pressure Determination of VM Using the Denuder-Liquid Chromatography-Mass Spectrometry Technique*; ECBC-TR-1278; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2015; UNCLASSIFIED Report (ADA613614).

28.   Buchanan, J.H.; Sumpter, K.B.; Abercrombie, P.L.; Tevault, D.E. *Vapor Pressure of GB*; ECBC-TR-686; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2009; UNCLASSIFIED Report (ADA500820).

29.   Williams. B.R.; Hulet, M.S.; Samuels, A.C.; Miles, R.W., Jr. *Vapor-Phase Infrared Spectral Study of Weapons-Grade O-Ethyl S-2-(Diisopropylamino)ethyl Methylphosphonothiolate (VX)*; ECBC-TR-955; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2012; UNCLASSIFIED Report (ADA562293).

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ACRONYMS AND ABBREVIATIONS

CAS	Chemical Abstracts Service
C_{sat}	saturation concentration; also referred to as volatility
CWA	chemical warfare agents
DICDI	*N,N′*-diisopropylcarbodiimide
DSC	differential scanning calorimeter
ECBC	U.S. Army Edgewood Chemical Biological Center
GB	isopropyl methylphosphonofluoridate
GC	gas chromatography
ΔH_{vap}	enthalpy of vaporization
LC	liquid chromatography
M	molecular mass
MS	mass spectrometry
n_a	moles of analyte
n_{car}	moles of carrier gas
P	vapor pressure
P_{amb}	ambient atmospheric pressure
P_{calc}	calculated vapor pressure
P_{expt}	experimental vapor pressure
ΔS_{vap}	entropy of vaporization
T	temperature
T_C	temperature in Celsius
T_K	temperature in Kelvin
T_{NBPt}	normal boiling point temperature
VX	*O*-ethyl *S*-(2-diisopropylaminoethyl) methyl phosphonothioate

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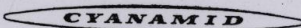
CYANAMID

AMERICAN CYANAMID COMPANY
INTERMEDIATES DEPARTMENT
BOUND BROOK, NEW JERSEY
ELLIOT 6-2000

September 13, 1962

Mr. H. V. Rouse, Jr.
U. S. Army CBR Engineering Group
Army Chemical Center
Maryland

Dear Mr. Rouse:

Following our telephone conversation last week, I found that our laboratories had determined vapor pressures of diisopropyl carbodiimide. These data are as follows:

Vapor Pressure, Millimeters	Temperature °C.
20	51
50	71
100	87
200	105
400	125
(760)	(146)

The above values were read from a smooth curve on semi-log paper. Determinations were made in the range of 50 to 125°C., and the normal boiling point shown above was obtained by extrapolation.

The measurements were carried out in a conventional isoteniscope; pressures were read to two significant figures; and temperatures were read to the nearest half degree. While no warranty is expressed or implied regarding the precision of these data, we believe they are sufficiently accurate for engineering purposes.

I find that we have measured no other physical properties of diisopropyl carbodiimide.

On the day of our conversation, I received a copy of Dr. Levinskas' letter to you outlining a proposed toxicity testing program on diisopropyl carbodiimide, and I assume your copy has reached you by this time.

If we can assist you further, please call on us.

Sincerely yours,

Edward Groth, Jr.
Manager, Sales Development

EGJr/jc

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