

Full Paper

Knudsen Effusion Measurement of Organic Peroxide Vapor Pressures

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Abstract

The vapor pressures of TATP over the temperature range 269.85–306.95 K and DADP over the temperature range 265.85–294.85 K were determined using a modified Knudsen effusion apparatus. The Clausius-Clapeyron plot of $\log_{10}(p(\text{Pa}))$ with $1/T$ provided a straight line for each material. This expression for TATP is $\log_{10}(p(\text{Pa})) = -(4497 \pm 80)/T(\text{K}) + (15.86 \pm 0.28)$ (error limits are 95% confidence limits) and for DADP it is $\log_{10}(p(\text{Pa})) = -(4417 \pm 137)/T(\text{K}) + (16.31 \pm 0.48)$. These expressions yield values of the vapor pressure at 298.15 K of 6 Pa for TATP and 17 Pa for DADP, and heats of sublimation of $86.2 \pm 1.5 \text{ kJ mol}^{-1}$ for TATP and $84.6 \pm 2.6 \text{ kJ mol}^{-1}$ for DADP. Attempts were made to determine the vapor pressure of HMTD but it appears to have a vapor pressure too low for our system to reliably determine. A two month experiment did provide an upper limit estimate for the vapor pressure of HMTD of approximately 0.04 Pa at room temperature. Melting point and melting point range were used as verification of the identity and purity of the TATP and DADP used in these experiments, but this was not possible with HMTD since it detonates prior to melting.

Keywords: Diacetone Diperoxide, Explosive Detection, Hexamethylene Triperoxide Diamine, Triacetone Triperoxide, Vapor Pressure of Explosives

1 Introduction

Triacetone triperoxide (TATP) is the most common member of a developing class of peroxide-based explosives. It is a primary explosive, and because of its sensitivity and instability it has no commercial or military applications. On the other hand, it can be easily made from commonly available ingredients [1]. Some of the peroxide-based explosives, including TATP and diacetone diperoxide (DADP), have high vapor pressures, even as they lack some of the attributes used to detect traces of

other common explosives. Also in this general class is the less-sensitive and less-volatile hexamethylene triperoxide diamine (HMTD). The exemplary status of these compounds makes accurate determinations of their vapor pressures of particular importance. The potential for reaction and decomposition of these unstable compounds suggests that a variety of experimental methods should be used to guard against experimental artifacts in any one technique. In this work we present results from the classical Knudsen effusion technique [2–5], which turns out to be well-suited to measuring vapor pressures in the high range exhibited by TATP and DADP (and relatively ill-suited to measuring the lower vapor pressure of HMTD). To validate our experimental technique, vapor pressure measurements were also made of naphthalene, producing results in good agreement with literature values.

2 Experimental Part

2.1 Synthesis of TATP, DADP, and HMTD

TATP was prepared by the acid resin method. Eight milliliters of acetone was placed in a 100 mL beaker which in turn was placed in a thermos container holding acetone which had been cooled to -40°C by the addition of dry ice. Additional dry ice was added to maintain the temperature around -40°C . A magnetic stir bar was placed in the acetone and 6.4 mL of 50% w/w H_2O_2 was slowly added to the beaker and mixed with the acetone. Several grams of strongly cationic acid resin, Dowex HCR-W2 (strongly acid, 16–40 mesh, Aldrich 42.870-1), was washed with water as recommended by the supplier. Four grams of this washed resin was added to the stirring mixture. The mixture was allowed to gradually warm to room temperature and was continually stirred for 10–12 h. Forty

milliliter of CH_2Cl_2 was added to the paste to dissolve the product. The solution was filtered to remove the acid resin and transferred to a Petri dish to allow the solvent to evaporate. The white solid produced was dissolved in warm methanol and recrystallized from the cooled methanol solution.

Comparison of the above TATP product was made to another (older) sample prepared by a modification of the acid-catalyzed synthesis proposed in 1959 by Milas and Golubovic [6]. A mixture of 5.6 mL acetone and 2.3 mL of 50% w/w H_2O_2 was cooled to -15°C . Four milliliters of conc. HNO_3 was added very slowly so that the solution remained cooler than 5°C . After the acid addition, the solution was kept at 0°C for 45 min. The resulting solution was placed into a separatory funnel along with 25 mL water and 40 mL CH_2Cl_2 . The water layer was removed and the remaining CH_2Cl_2 layer solution was washed again with 50 mL of water. The CH_2Cl_2 layer was collected and dried overnight with molecular sieve. The dried CH_2Cl_2 solution was poured into a petri dish from which the solvent could evaporate. White crystals of TATP remained after evaporation of the CH_2Cl_2 . The TATP was recrystallized from warm methanol.

The melting points of the two samples of TATP, the acid resin sample prepared for this work in September of 2006, and the acid catalyzed sample prepared in 1996, were measured. Melting points for both samples as well as a mixed melting point of the two samples were in the range of $94.2\text{--}95.2^\circ\text{C}$.

DADP was prepared by a method similar to that described by Dubnikova et al. [7]. A methylene chloride solution of acetone with a trace of methanesulfonic acid was cooled to -10°C and an equivalent amount of concentrated (50 to 70%) hydrogen peroxide was slowly added. After 1 h, this mixture was added to a separatory funnel, the methylene chloride solution was separated from the aqueous spent peroxide and the acid dissolved in it. If the methylene chloride layer is allowed to evaporate, there is no residue. The methylene chloride was treated again with a significant amount of acid, at 0°C . After an hour, the methylene chloride layer was washed several times with water. The organic layer was separated and allowed to evaporate. The crude DADP was recrystallized from hot methanol. The melting point was determined to be $138\text{--}139.2^\circ\text{C}$.

Our preparations of TATP and DADP are consistent with results reported by Matyas et al. [8], who studied the stability of TATP and found the acid catalyst to be an important consideration. When nitric acid is used as the catalyst for the preparation of TATP after 2 months only a trace amount of DADP was found. However, when TATP is prepared using methanesulfonic acid as a catalyst it transforms to DADP in a comparatively rapid manner.

HMTD was prepared by a procedure modified from that described by Schaefer et al. [9]. Five milliliter of 30% H_2O_2 was placed in a beaker set in an ice bath at 0°C . Hexamethylenetetramine (1.4 g) was slowly added to the peroxide solution. The peroxide solution was al-

lowed to cool back to 0°C . Citric acid (2.1 g) was slowly added to the peroxide solution. The solution was allowed to remain in the ice bath for 3 h. After this time, the beaker was transferred to a 15°C water bath for 12 h. The precipitated HMTD crystals were filtered off with a paper filter, washed with deionized water, and then with ethanol. **WARNING!** No melting point determination can be made with this material as it detonates prior to melting. Detonation occurred as low as 73°C and as high as 147°C . The low value was associated with a freshly prepared sample of HMTD (24 h old) while higher values were repeatedly observed in samples allowed to stand for 2 weeks. Wierzbicki and Cioffi [10] report a melting point of $151\text{--}152^\circ\text{C}$ (dec) while other literature melting point values from 144 to 145°C [11] to 154°C [12] have been reported.

2.2 Apparatus

The Knudsen reaction vial was constructed from a Chem-glass, Inc., 10 mL conical thin-walled, reaction vial with 14/10 threaded outer joint (MW-54-05) which was then glassblown to configure it into a round bottom 8 mL vial. This vial was supplied with a plastic screw cap and septum of silicone rubber with one face of Red Teflon and the other of an ultrathin coating of polypropylene. When used with Viton O-rings and threaded outer joints these caps provide a vacuum tight seal. A 12 mm diameter hole was cut in the septum, and an 18 mm diameter disk of heavy duty aluminum foil (thickness 0.023 mm) was placed between the Teflon surface of the septum and the top of the vial.

Orifices were made using an alignment fixture and a 0.338 mm diameter drill bit held in a pin vise. Microscopic images of the orifices and image analysis software were used to determine the actual orifice areas, which were typically in the range of $0.14\text{--}0.18\text{ mm}^2$. This procedure provided a reproducible orifice in the aluminum disk with a flat surface on the side toward the sample under study with any irregularities created being restricted to the vacuum side of the disk. The Clausing factor [13,14], which multiplies the physical area to yield an effective orifice area, is about 0.95 for the above values of orifice size and foil thickness.

A thermal transfer chamber was constructed using a closed end stainless steel tube approximately 11 cm long with an OD of 2 cm and an ID of 1.8 cm. This tube was silver soldered into a stainless steel O-ring joint (Kontes® Cat. No. 674850-0025) such that 5 cm remained exterior to the joint body and 6 cm interior. A tube was made by wrapping layers of aluminum foil around the reaction vial so that it would fit snugly into the stainless steel tube of the thermal transfer apparatus. This aluminum tube remained as a stationary part of the thermal transfer assembly and provided for good thermal contact between the reaction vial and the main body of the assembly when thermal contraction and expansion took place in the system. A thin copper wire tether attached to the reaction

vial allowed for easy removal of the vial from the aluminum tube. After the reaction vial was placed into the thermal transfer assembly a series of steel washers of increasing weight was placed on top of the vial to provide thermal contact between the surface of the orifice disk and the stainless steel assembly. Connection between the thermal transfer chamber and the vacuum system was made through a glass O-ring joint adapter and a 12 inch Cajon flexible assembly.

The temperature of the system was maintained by the fluid from a Neslab Refrigerated Bath Circulator (Model RTE-110) which was pumped through a copper coil placed in a 10 L insulated plastic bucket. The ethylene glycol/water fluid in the plastic bucket was stirred by a submersible pump with an output of 20 L min⁻¹, modified to have four 0.63 cm diameter outlets to act as a circulator bath. The temperature of the bath fluid was determined using a Fluke 51 II thermometer with a type K (chromel/alumel) thermocouple. The temperature was verified by standardization with a National Bureau of Standards (NIST) certified thermometer system. This measurement system provided a temperature sensitivity of 0.1 °C. Once an equilibrium temperature was attained, no temperature variation was observed over the run times.

The Knudsen cell system was thermostated by placing the reaction vial into the thermal transfer chamber, attaching the O-ring adapter to the chamber and flexible Cajon assembly, and placing the thermal transfer chamber into a 1 gallon zip-lock freezer bag. The stainless steel portion of the thermal transfer assembly could then be submerged to a level half way up the glass O-ring adapter without having any bath fluid come in contact with the surface of the thermal transfer assembly. Tests showed the thermal transfer through the plastic film to be sufficient for the purposes of this study.

The vacuum system was constructed from glass O-ring connectors and Teflon stopcock valve components. The vacuum was provided by an Edwards two stage direct drive vacuum pump (Model E2M8). The pressure of the evacuated system was determined by an Edwards Pirani gauge Model 1001 and a PRM-10 probe. The vacuum assembly was capable of reducing the pressure of the system to <0.1 Pa. Mass determinations were made using a Mettler Toledo Balance, Model AB/135-S/FACT which has a readability of 0.01 mg and a repeatability of ± 0.03 mg for mass quantities <31 g. Time intervals were measured using a stopwatch. Melting points were determined using a Barnstead International Electrothermal Mel-Temp®.

2.3 Validation of Experimental Procedures

All data reported here use the bath temperature as the sample temperature within the reaction vial. This was validated by preliminary experiments in which molecular sieve material was placed in the reaction vial before it was placed into the thermal transfer assembly and then

into the fluid bath. The temperature of the molecular sieve was measured using a type K thermocouple, protected within a thin walled glass tube, and the Fluke thermometer. The comparison to the temperature of the bath was run over the range 1–38 °C. In all cases the temperatures agreed within ± 0.1 °C after approximately 45 min of submersion. This minimum equilibration time was observed for all data points.

Given this initial equilibration time, carried out with the entire system still at atmospheric pressure, it is important to verify that no sample is lost during the thermostating process. A vial with a TATP sample was placed in the reaction system with the escape orifice in place as it would be during a Knudsen effusion run. The pressure on both sides of the escape orifice disk was left at one atmosphere and the assembly was heated to 40 °C for 1 h. The vial and sample were then removed, allowed to cool, and weighed. The mass of vial and sample was found to be unchanged, as is expected given the slow rate of diffusion at atmospheric pressure. A similar experiment using DADP produced an identical result. This allowed the establishment of a consistent procedure for time measurement: the start time was when the thermal transfer chamber external to the Knudsen cell was evacuated, and the stop time was when the atmospheric pressure was allowed to enter the thermal transfer chamber.

Verification of the reliability of the effusion system as a means of determining vapor pressure was obtained by comparing our determination for the vapor pressure of naphthalene with published literature values. Naphthalene was the material of choice as it has a well documented value for its vapor pressure, can be purchased at high purity, and has an ambient vapor pressure of similar magnitude to those determined for TATP and DADP. The naphthalene of 99.6% purity used in our determination was obtained from Alfa Aesar.

3 Results and Discussion

3.1 Validation Measurements of Naphthalene

Two measurements of the vapor pressure of naphthalene at 21.8 °C resulted in a mean value of 7.72 Pa (0.0585 Torr). Interpolation of the data presented by Ruzicka et al. [15] yields a vapor pressure at 21.8 °C of 7.87 Pa (0.0596 Torr). This difference of 0.15 Pa can be viewed in the context of estimates of possible random and systematic errors. As to the former, we can cite the 0.053 Pa difference between our two measurements, and for the latter, we note that the entire 0.15 Pa difference could be accounted for by a difference of 0.2 °C between the temperature of the thermal bath and the temperature of the crystalline sample.

3.2 Measurements of TATP

A Clausius-Clapeyron data plot for TATP, using data presented in Table 1, is displayed in Figure 1. Values for the

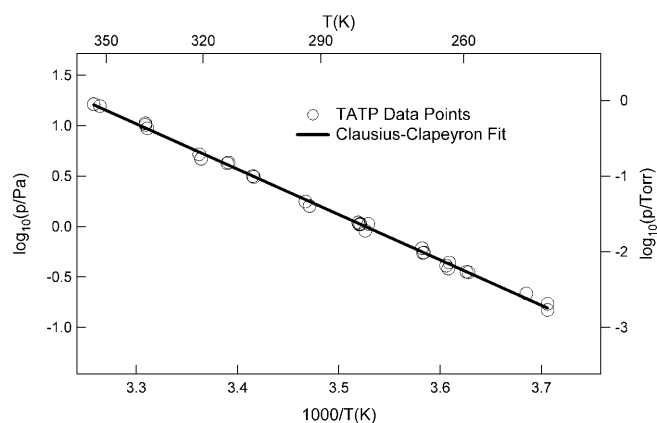


Figure 1. TATP vapor pressure data points and fit to Clausius-Clapeyron equation.

vapor pressure were determined over the temperature range 269.85–306.95 K (–3.3–33.8 °C). The data fit the expression: $\log_{10}(p(\text{Pa})) = -(4497 \pm 80)/T(\text{K}) + (15.86 \pm 0.28)$ where the error bounds are 95 % confidence limits derived from the scatter in the data points. (For the purpose of comparison to earlier reports, this expression can be converted to $\log_{10}(p(\text{Torr})) = -(4497 \pm 80)/T + (13.74 \pm$

0.28). The R^2 value for the fit is 0.9977. The slope of the graph corresponds to a value of $86.2 \pm 1.5 \text{ kJ mol}^{-1}$ ($20.6 \text{ kcal mol}^{-1}$) for the heat of sublimation of TATP. Evaluating the Clausius-Clapeyron fit expression at 25 °C yields a value of 6.0 Pa (0.045 Torr).

In Figure 2 we compare the data reported here with previous measurements. Five published measurements of TATP vapor pressures are known to us (more have been made but are currently not generally available). However, of the five, two used spectroscopic techniques that provide only relative vapor pressures as a function of temperature. Oxley et al. [16] used a gas chromatographic technique over the temperature range 285–333 K. They reported a value of about 7 Pa (0.052 Torr) at 298 K, and a Clausius-Clapeyron fit to their data yielding a value of the heat of sublimation reported at various points in the paper as 109 and 104 kJ mol^{-1} . Ramírez et al. [17] made thermogravimetric observations, calibrated using benzoic acid, over the temperature range of 298–348 K. They reported a value of about 2.56 Pa (0.0192 Torr) at 298 K, and a heat of sublimation from a Clausius-Clapeyron fit to their data of 85.8 kJ mol^{-1} .

Cavity ringdown spectroscopy measurements, reported in Todd et al. [18] and Steinfeld et al. [19], of the infrared absorption cross section of the 7.3 and 7.8 μm absorption

Table 1. Vapor pressures versus temperature for TATP.

T (K)	Mass Loss (g)	Time (s)	Orifice Area (10^{-3} cm^2)	Vapor Pressure (Pa)	$\log_{10}(p)$	1000/T (K^{-1})
269.85	0.00286	30895	1.684	0.14932	–0.826	3.706
269.85	0.00348	32605	1.684	0.17198	–0.765	3.706
271.45	0.00333	26982	1.541	0.21732	–0.662	3.685
275.65	0.00454	20985	1.684	0.35197	–0.453	3.628
275.85	0.00510	23176	1.684	0.35730	–0.446	3.626
277.15	0.00351	14149	1.541	0.44130	–0.354	3.609
277.25	0.00420	19498	1.541	0.38397	–0.415	3.608
277.35	0.00347	15081	1.541	0.41063	–0.387	3.606
279.05	0.00120	3689	1.621	0.55329	–0.257	3.584
279.15	0.00127	3930	1.621	0.54929	–0.260	3.583
279.25	0.00383	10259	1.684	0.61062	–0.214	3.582
283.45	0.00239	4182	1.492	1.06391	0.0269	3.529
283.65	0.00159	3256	1.492	0.90926	–0.0411	3.526
284.05	0.00498	7821	1.684	1.05058	0.0219	3.521
284.05	0.00467	7219	1.684	1.06791	0.0289	3.521
284.15	0.00457	6319	1.919	1.04791	0.0209	3.520
284.25	0.00523	6887	1.919	1.09991	0.0419	3.519
288.15	0.00255	3001	1.492	1.59454	0.2029	3.471
288.15	0.00278	3261	1.492	1.59987	0.2039	3.471
288.45	0.00486	4566	1.684	1.77052	0.2479	3.467
292.75	0.00600	3529	1.545	3.10508	0.4919	3.416
292.85	0.00802	4628	1.545	3.16507	0.5009	3.415
294.95	0.01860	8573	1.423	4.31831	0.6359	3.391
295.05	0.01919	9024	1.423	4.23299	0.6269	3.390
297.35	0.01716	5806	1.613	5.21024	0.7169	3.364
297.45	0.00621	2519	1.492	4.69961	0.6719	3.362
302.05	0.00634	1295	1.492	9.40456	0.9739	3.311
302.25	0.01123	2119	1.492	10.1832	1.0079	3.309
302.25	0.00646	1176	1.492	10.5551	1.0239	3.309
306.45	0.02439	3161	1.423	15.6534	1.1948	3.264
306.95	0.01159	1274	1.613	16.2947	1.2122	3.258
306.95	0.01662	1820	1.613	16.3573	1.2139	3.258

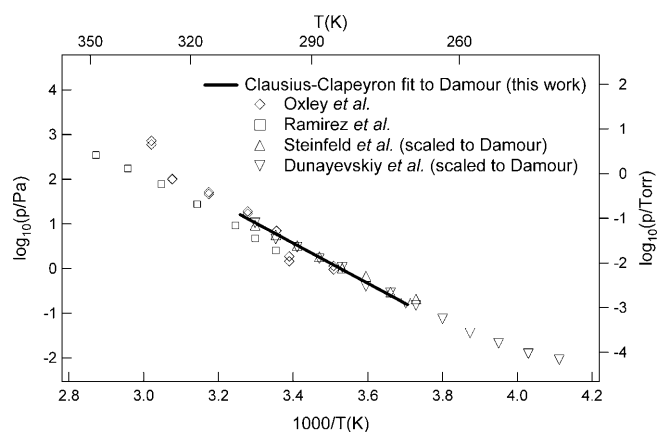


Figure 2. TATP fit expression from this work, compared to previously published data.

bands in the temperature range 268.15–303.15 K yielded one of the two relative data sets shown in the graph. Dunayevskiy et al. [20] used photoacoustic spectroscopy with a tunable infrared quantum cascade laser to make measurements over the temperature range 243.15–303.15 K. In Figure 2, the Steinfeld et al. and Dunayevskiy et al. data points have been scaled so that the Clausius-Clapeyron line fit through them passes through the Damour curve at ambient temperature.

The work of Dunayevskiy et al. is a high precision data set that extends to much lower temperatures than any other study. However, the presence of some systematic error is clear from the non-physical [21] upward curvature of their low temperature points. Obviously, the heat of sublimation derived from a Clausius-Clapeyron fit to this data set will increase as more low temperature points are excluded. It turns out that although Dunayevskiy et al. report a heat of sublimation of 86 kJ mol^{-1} , our fit to their transcribed data points yields a value above 80 kJ mol^{-1} only if we exclude the lowest 5 data points. With the possibility of different weightings of the literature data sets in mind, we present in Table 2 the results of our analysis of the five data sets with equal weighting of all reported points (the error limits are 95 % confidence limits derived from our fits).

Table 2. Thermodynamic parameters from our analysis of reported TATP vapor pressure data sets^{a)}.

	$\Delta_{\text{sub}}H$ (kJ mol^{-1})	Vapor Pressure at 298.15 K (Pa)
Damour et al. (this work)	86.2 ± 1.5	6.0
Oxley et al. [16]	104.5 ± 13.0	5.7
Ramirez et al. [17]	85.8 ± 5.9	2.8
Steinfeld et al. [19]	75.1 ± 4.7	
Dunayevskiy et al. [20]	81.3 ± 2.6	

^{a)} Some parameters are derived from our fits to tabulated data, and may not agree with values presented in the original papers.

3.3 Measurements of DADP

The data and Clausius-Clapeyron graph for DADP are displayed in Table 3 and Figure 3, respectively. Values for the vapor pressure were determined over the temperature range 265.85–294.85 K (-7.3 to $+21.7^\circ\text{C}$). The fit expression is given by $\log_{10}(p(\text{Pa})) = -(4417 \pm 137)/T(\text{K}) + (16.31 \pm 0.48)$ (for comparison, converting to $\log_{10}(p(\text{Torr})) = -(4417 \pm 137)/T + (14.19 \pm 0.48)$) where the error bounds are 95 % confidence limits derived from the scatter in the data points. The R^2 value for the fit is 0.9988. The slope of the graph corresponds to a value of $84.6 \pm 2.6 \text{ kJ mol}^{-1}$ ($20.2 \text{ kcal mol}^{-1}$) for the heat of sublimation of DADP. Evaluating the Clausius-Clapeyron fit expression at 25°C yields a value of 17 Pa (0.13 Torr).

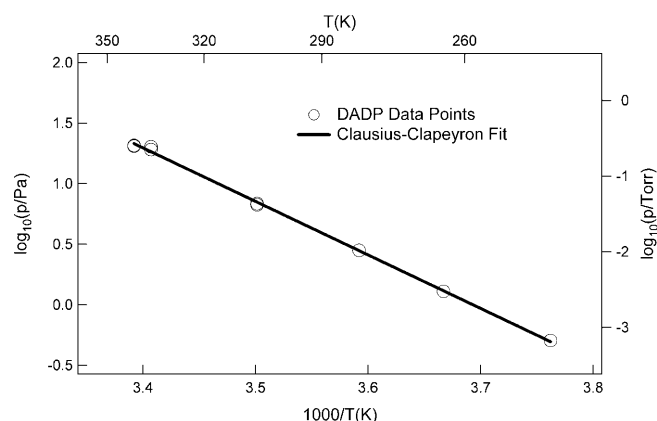


Figure 3. DADP vapor pressure data points and fit to Clausius-Clapeyron equation.

3.4 Measurements of HMTD

As mentioned above, all attempts to measure a melting point resulted in rapid decomposition of the sample. The first sample, air-dried overnight, detonated at 73.7°C while subsequent samples, measured after both shorter and longer drying times, decomposed around 150°C . Reasons for these different behaviors have not been established. We were also unsuccessful in our attempts to find an appropriate solvent to use for purification of the HMTD by recrystallization. It appears the HMTD has only very limited solubility, at best, in all solvents tested (from polar to non-polar, including water, methanol, dimethylsulfoxide, dichloromethane, acetone, diethyl ether, toluene, and benzene).

Attempts to carry out conventional Knudsen effusion experiments resulted in HMTD mass losses at the lower limit of reliability of our balance (i.e., 0.00002 g). One additional attempt was made by comparing mass losses at atmospheric pressure for samples of HTMD and TATP. Both sample vials had escape orifices of about 0.16 mm^2 , were set at room temperature, and were open to 1 atmosphere pressure through the orifice. They were left in these conditions for approximately 2 months. After that

Table 3. Vapor pressures versus temperature for DADP.

T (K)	Mass Loss (g)	Time (s)	Orifice Area (10 ⁻³ cm ²)	Vapor Pressure (Pa)	log ₁₀ (p)	1000/T (K ⁻¹)
265.85	0.01113	44494	1.623	0.50663	-0.2941	3.762
272.75	0.01050	16709	1.623	1.29323	0.1109	3.667
278.45	0.01070	7883	1.623	2.81310	0.4499	3.592
285.65	0.00983	3013	1.623	6.86610	0.8369	3.501
285.65	0.00827	2592	1.623	6.70612	0.8269	3.501
293.55	0.01612	1700	1.623	20.2117	1.3058	3.407
293.55	0.01080	1201	1.623	19.1718	1.2828	3.407
294.85	0.01726	1803	1.623	20.4517	1.3109	3.392
294.85	0.00900	928	1.623	20.7183	1.3165	3.392

time the mass loss by TATP was 0.00408 g and the mass loss by HMTD was 0.00003 g. The calculated vapor pressure for HMTD from this trial is 0.04 Pa (3×10^{-4} Torr). This mass loss is still at the lower limit of reliability of our balance. Therefore, we can only say that the vapor pressure of HMTD is very low compared to TATP and DADP.

(Note added in proof: We thank the referee for drawing our attention to reference [22], which contains similar results for TATP and DADP and reports that the HMTD vapor pressure was not determined due to its low volatility and its rapid thermal decomposition at low temperatures).

4 Conclusion

We have presented vapor pressure measurements of TATP resulting in a value at ambient conditions (25 °C) of 6 ± 0.3 Pa (error bound is a 95% confidence limit based only on scatter in the data set). This is much higher than vapor pressures of other common explosives, and the 25 °C value from our study of DADP of 17 Pa is higher still. We could not measure a value of HMTD vapor pressure, which could be far below the upper limit reported here of approximately 0.04 Pa.

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