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The enthalpies of formation of (dimethylamino)dichlorophosphine, bis(dimethylamino)chlorophosphine, and tris(dimethylamino)phosphine

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The standard molar enthalpies of formation at the temperature 298.15 K of $(CH_3)_2NPCl_2(l)$, $\{(CH_3)_2N\}_2PCl(l)$, and $\{(CH_3)_2N\}_3P(l)$ have been determined by reaction calorimetry as $-(286.3 \pm 2.4)\ kJ \cdot mol^{-1}$, $-(220.1 \pm 2.3)\ kJ \cdot mol^{-1}$, and $-(112.8 \pm 2.3)\ kJ \cdot mol^{-1}$, respectively. Enthalpies of vaporization have been obtained from vapour-pressure measurements and the corresponding values of $\Delta_f H_m^\circ(g)$ derived as $-(245.5 \pm 2.5)\ kJ \cdot mol^{-1}$, $-(174.2 \pm 2.6)\ kJ \cdot mol^{-1}$, and $-(71.3 \pm 2.4)\ kJ \cdot mol^{-1}$, respectively. The values of the bond enthalpies D(P–Cl) and D(P–N) in substituted phosphines are discussed.

1. Introduction

The compounds $\{(CH_3)_2N\}_v PCl_{3-v}$, $v = 0, 1, 2, 3$, all undergo rapid stoichiometric hydrolysis in acidic media to yield well defined products with known enthalpies of formation. Their enthalpies of formation are, therefore, readily determined by solution-reaction calorimetry and the results for the series of four compounds provide information on the enthalpies of the (P–Cl) and (P–N) bonds in substituted phosphines. This paper reports thermochemical measurements on three members of the series ($v = 1, 2, 3$) for which results have not previously been published. Enthalpies of vaporization at the temperature $T = 298.15$ K of the three compounds were derived from vapour-pressure measurements.

2. Experimental

(Dimethylamino)dichlorophosphine was prepared according to the procedure described by Morse *et al.*[1] Anhydrous dimethylamine vapour was condensed on to a cold finger and the liquid allowed to drip into a chilled stirred ethereal solution

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of PCl_3. The reaction mixture was fractionally distilled and the fraction boiling between $T = 420$ K and 423 K collected. The product was re-distilled and the purity estimated by determination of the chloride ion (Volhard's method) after hydrolysis with dilute nitric acid and oxidation of the H_3PO_3 with H_2O_2 (to avoid reduction of the $AgNO_3$ by H_3PO_3 to silver). Mass fraction of Cl: found, 0.485; calculated for $\{(CH_3)_2N\}PCl_2$, 0.489.

Bis(dimethylamino)chlorophosphine was prepared (i), by the reaction between tris(dimethylamino)phosphine (Aldrich) and PCl_3 in ethereal solution;(2) and (ii), by the addition of PCl_3 to dimethylamine in ethereal solution. In both cases the reaction mixture was distilled under dry nitrogen and the fraction boiling between $T = 331$ K and 335 K (pressure $p = 1.27$ kPa) collected. The purity of the re-distilled product was estimated by determination of the chloride ion produced by acid hydrolysis (see above). Mass fraction of Cl: found, 0.229; calculated for $\{(CH_3)_2N\}_2PCl$, 0.230.

Tris(dimethylamino)phosphine was prepared by the reaction between PCl_3 and excess anhydrous $(CH_3)_2NH$ in ethereal solution,(3) and was also obtained from Aldrich. It was purified by distillation and the fraction boiling at $T = 435$ K to 437 K ($p = 101.325$ kPa) collected. The purity was estimated by hydrolysis with excess HCl, followed by potentiometric titration of the hydrolysate with standard NaOH(aq). The difference between the first and second end-points enabled the phosphorus content to be determined (as H_3PO_3). Mass fraction of P: found 0.189; calculated for $\{(CH_3)_2N\}_3P$, 0.190.

A differential twin-vessel reaction calorimeter, operator in the isoperibol mode at $T = 298.15$ K, was employed.(4) Its performance was frequently checked by measurements of the enthalpy of neutralization of tris(hydroxymethyl)-aminomethane (THAM) with excess $0.1\ \text{mol}\cdot\text{dm}^{-3}$ HCl(aq). Because of the sensitivity of the compounds to air and moisture, the glass ampoules were filled in a dry-box containing nitrogen.

The sample for vapour-pressure measurements was contained in a glass tube surrounded by a jacket, through which water thermostatted to ± 0.02 K was circulated, and connected to a strain-gauge (Schaevitz EM Ltd, model P522-0001, range of pressure: 0 to 34.7 kPa) and high vacuum system. To prevent condensation of vapour, all parts of the apparatus outside the thermostatted jacket were electrically heated to a temperature above that of the thermostat. The apparatus was tested by measurements of the vapour pressure of conductivity water between $T = 278$ K and 318 K; the mean deviation of nine values from those recommended by IUPAC(5) was (0.37 ± 0.34) per cent.†

3. Results

The hydrolysis of the three substituted phosphines was carried out using $2\ \text{mol}\cdot\text{dm}^{-3}$ HCl(aq). Preliminary experiments with $\{(CH_3)_2N\}_3P$, the phosphine most resistant to hydrolysis, indicated that the reaction was complete within the main period of a calorimetric experiment (about 10 min). This was ascertained by

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† Throughout this paper uncertainty intervals of reported experimental results denote 95 per cent confidence limits, calculated using Student's t at the appropriate number of degrees of freedom.

TABLE 1. Hydrolysis of a mass m of $(CH_3)_2NPCl_2(l)$ in a volume 100 cm^3 of 2 mol·dm^{-3} HCl(aq) $\triangleq$ $0.2(HCl + 27.76H_2O)$ at $T = 298.15$ K

m/g [a]	$-\Delta_r H_m$/(kJ·mol^{-1}) (observed)	$-\Delta H_m$/(kJ·mol^{-1}) (dilution)	$-\Delta_r H_m$/(kJ·mol^{-1}) (corrected)
0.08093	254.66	2.02	256.68
0.07992	254.10	2.05	256.15
0.07552	254.10	2.04	256.14
0.05811	253.27	2.06	255.33
0.05807	252.97	2.06	255.03
0.04991	254.81	2.05	256.86
0.04124	253.63	2.06	255.69
0.04047	252.95	2.0	255.00
0.02727	253.38	2.12	255.50
0.02290	253.73	2.10	255.83
		Mean:	255.82 (±0.45)

[a] Weighings in air corrected using $\rho/(g\cdot cm^{-3}) = 1.35$ (estimated from the values for PCl_3 and $\{(CH_3)_2N\}_3P$, where ρ denotes density.

calculating the cooling constant after different main periods and only at 10 min was the cooling constant identical to that obtained for an electrical calibration.

The stoichiometry was established by determination of Cl^- (using Volhard's method) and H_3PO_3 (potentiometrically) in the products. The general equation is

$$\{(CH_3)_2N\}_v PCl_{3-v}(l) + \{(2v - 3 + m)HCl + (p + 3)H_2O\} =$$
$$\{v(CH_3)_2NH_2Cl + H_3PO_3 + mHCl + pH_2O\},$$

and the results obtained for the three compounds are listed in tables 1 to 3. Because HCl is either produced ($v = 1$) or consumed ($v = 2, 3$) in the reaction and water is always consumed, these tables include a dilution or concentration term which is to be added to the observed reaction enthalpy to give the value which corresponds

TABLE 2. Hydrolysis of a mass m of $\{(CH_3)_2N\}_2PCl(l)$ in a volume 100 cm^3 of 2 mol·dm^{-3} HCl(aq) $\triangleq$ $0.2(HCl + 27.76H_2O)$ at $T = 298.15$ K

m/g [a]	$-\Delta_r H_m$/(kJ·mol^{-1}) (observed)	ΔH_m/(kJ·mol^{-1}) (concentration)	$-\Delta_r H_m$/(kJ·mol^{-1}) (corrected)
0.01588	280.87	1.52	279.35
0.01653	281.33	1.57	279.76
0.01818	280.22	1.63	278.59
0.02282	280.37	1.63	278.74
0.02911	280.78	1.66	279.12
0.03458	280.56	1.65	278.90
0.04729	280.48	1.65	278.83
0.05226	280.75	1.60	279.15
		Mean:	279.06 (±0.31)

[a] Weighings in air corrected using $\rho/(g\cdot cm^{-3}) = 1.060$.(2)

TABLE 3. Hydrolysis of a mass m of $[(CH_3)_2N]_3P(l)$ in a volume $100\,cm^3$ of $2\,mol\cdot dm^{-3}$ HCl(aq) $\triangleq$ $0.2(HCl + 27.76H_2O)$ at $T = 298.15$ K

m/g [a]	$-\Delta_rH_m/(kJ\cdot mol^{-1})$ (observed)	$\Delta H_m/(kJ\cdot mol^{-1})$ (concentration)	$-\Delta_rH_m/(kJ\cdot mol^{-1})$ (corrected)
0.01793	348.69	5.29	343.40
0.02096	348.76	5.34	343.42
0.02247	348.76	5.18	343.58
0.03930	348.76	5.26	343.50
0.04638	348.74	5.30	343.44
0.04673	348.72	5.34	343.38
0.04978	348.72	5.32	343.40
0.05059	348.69	5.28	343.41
			Mean: 343.44 (±0.06)

[a] Weighings in air corrected using $\rho/(g\cdot cm^{-3}) = 0.898$.(15)

to solution of the products in $2\,mol\cdot dm^{-3}$ HCl(aq) $(HCl + 27.76H_2O)$. In order to calculate molar enthalpies of formation of the the phosphorus compounds from these results, molar enthalpies of solution of $(CH_3)_2NH_2Cl(cr)$ and of $H_3PO_3(cr)$ in $2\,mol\cdot dm^{-3}$ HCl(aq) were also measured (tables 4 and 5). Thus, neglecting any

TABLE 4. Molar enthalpy of solution of a mass m of $(CH_3)_2NH_2Cl(cr)$ in a volume $100\,cm^3$ of $2\,mol\cdot dm^{-3}$ HCl(aq) $\triangleq$ $0.2(HCl + 27.76H_2O)$ at $T = 298.15$ K

m/g	$n(H_2O)/n\{(CH_3)_2NH_2Cl\}$	$\Delta_{sol}H^\circ_m/(kJ\cdot mol^{-1})$
0.13646	3317	3.22
0.15928	2842	3.25
0.16610	2725	3.24
0.21284	2127	3.25
0.24243	1867	3.23
0.28222	1604	3.23
		Mean: 3.24 ± 0.01

TABLE 5. Molar enthalpy of solution of a mass m of $H_3PO_3(cr)$ in a volume $100\,cm^3$ of $2\,mol\cdot dm^{-3}$ HCl(aq) $\triangleq$ $0.2(HCl + 27.76H_2O)$ at $T = 298.15$ K.

m/g [a]	$n(H_2O)/n(H_3PO_3)$	$\Delta_{sol}H^\circ_m/(kJ\cdot mol^{-1})$
0.06443	7064	7.94
0.08205	5547	7.76
0.08299	5484	8.19
0.10501	4334	7.80
0.12148	3747	8.05
0.27669	1645	7.91
		Mean: 7.94 ± 0.17

[a] Weighings in air corrected using $\rho/(g\cdot cm^{-3}) = 1.651$.(16)

TABLE 6. Molar enthalpy of solution of a mass m of $(CH_3)_2NH_2Cl(cr)$ in a volume 100 cm^3 of water at $T = 298.15$ K.

m/g	$n(H_2O)/n\{(CH_3)_2NH_2Cl\}$	$\Delta_{sol}H_m^\circ$/(kJ·mol^{-1})
0.08009	5652	2.03
0.10042	4508	2.07
0.23930	1892	2.09
0.27493	1646	2.02
0.28477	1591	2.04
0.28542	1586	2.07
		Mean: 2.05 ± 0.03

enthalpy of mixing between $\{H_3PO_3$ in $(HCl + 27.76H_2O)\}$ and $\{(CH_3)_2NH_2Cl$ in $(HCl + 27.76H_2O)\}$, the enthalpy of the final state in all the hydrolysis experiments: $\{H_3PO_3$ and $(CH_3)_2NH_2Cl$ in $(HCl + 27.76H_2O)\}$ was, therefore, known. There was no evidence for significant enthalpies of dilution in any of the determinations and, although the values reported in tables 4 and 5 refer to more concentrated solutions than were obtained in the hydrolysis experiments, no corrections have been applied.

Although $\Delta_f H_m^\circ\{(CH_3)_2NH_2Cl$ in $2000H_2O\} = -287.16$ kJ·mol^{-1} is given in reference 6, the corresponding value for the crystalline compound is not listed.(6) It was necessary, therefore, to determine the molar enthalpy of solution of $(CH_3)_2NH_2Cl(cr)$ in water and the results obtained are presented in table 6. From the mean value:

$$\Delta_{sol}H_m\{(CH_3)_2NH_2Cl \text{ in } vH_2O, 1600 < v < 5600\} = (2.05 \pm 0.03)\ \text{kJ·mol}^{-1},$$

$\Delta_f H_m^\circ\{(CH_3)_2NH_2Cl$, cr, 298.15 K$] = -(289.21 \pm 0.03)$ kJ·mol^{-1}, neglecting the unknown uncertainty on the NBS value. The quantity $\Delta_f H_m^\circ(H_3PO_3,cr) = -(957.1 \pm 2.3)$ kJ·mol^{-1} was derived from the molar enthalpy of oxidation of $H_3PO_3(cr)$ to $H_3PO_4(aq)$,(7) $\Delta_f H_m^\circ(H_3PO_4$ in $4OH_2O)$,(8) and the enthalpy of dilution of $H_3PO_4(aq)$.(6) The enthalpy of formation of (HCl in $27.76H_2O$) was derived from $\Delta_f H_m^\circ(Cl^-,\infty) = -(167.08 \pm 0.10)$ kJ·mol^{-1},(9) and molar enthalpies of dilution(6) as $-(164.44 \pm 0.10)$ kJ·mol^{-1}. From the above ancillary quantities and the results reported in tables 1 to 5, the molar enthalpies of formation of the three phosphorus compounds were obtained and are listed in table 7.

Enthalpies of vaporization were derived from the linear plots (correlation coefficients, >99.8 per cent) of lg(p/Pa) against K/T. Because the mean temperatures of the measurements of the vapour pressure of $\{(CH_3)_2N\}_v PCl_{3-v}$ were close to

TABLE 7. $\Delta_f H_m^\circ(l)$, $\Delta_{vap}H_m^\circ(l)$, and $\Delta_f H_m^\circ(g)$ for $\{(CH_3)_2N\}_v PCl_{3-v}$ (v = 1, 2, 3) at $T = 298.15$ K

v	$-\Delta_f H_m^\circ(l)$ / kJ·mol^{-1}	$\Delta_{vap}H_m^\circ(l)$ / kJ·mol^{-1}	$-\Delta_f H_m^\circ(g)$ / kJ·mol^{-1}
1	286.3 ± 2.4	40.80 ± 0.68	245.5 ± 2.5
2	220.1 ± 2.3	45.91 ± 1.21	174.2 ± 2.6
3	112.8 ± 2.3	41.48 ± 0.58	71.3 ± 2.4

298.15 K (300 K, 310 K, 301 K for $v = 1, 2, 3$ respectively), no temperature corrections, which lay within the uncertainty intervals, were applied. The values of $\Delta_{\mathrm{vap}}H^{\circ}_{\mathrm{m}}(298.15\ \mathrm{K})$ and the derived values of $\Delta_{\mathrm{f}}H^{\circ}_{\mathrm{m}}(\mathrm{g})$ for the three compounds are listed in table 7.

4. Discussion

Of the three compounds for which molar enthalpies of formation have been determined, a value for only $\{(\mathrm{CH_3})_2\mathrm{N}\}_3\mathrm{P(l)}$ has been previously reported, based on the reaction between $(\mathrm{CH_3})_2\mathrm{NH(l)}$ and $\mathrm{PCl_3(l)}$.(10) This value: $-184.4\ \mathrm{kJ \cdot mol^{-1}}$, differs considerably from that obtained in the present study: $-(112.8 \pm 2.3)\ \mathrm{kJ \cdot mol^{-1}}$. An independent estimate of $\Delta_{\mathrm{f}}H^{\circ}_{\mathrm{m}}[\{(\mathrm{CH_3})_2\mathrm{N}\}_3\mathrm{P},\ \mathrm{g}]$ can be obtained from the relation:

$$\Delta_{\mathrm{f}}H^{\circ}_{\mathrm{m}}[\{(\mathrm{CH_3})_2\mathrm{N}\}_3\mathrm{P},\ \mathrm{g}] = \Delta_{\mathrm{f}}H^{\circ}_{\mathrm{m}}[\{(\mathrm{C_2H_5})_2\mathrm{N}\}_3\mathrm{P},\ \mathrm{g}] - 3[\{\Delta_{\mathrm{f}}H^{\circ}_{\mathrm{m}}(\mathrm{C_2H_5})_2\mathrm{NH},\ \mathrm{g}\} - \{\Delta_{\mathrm{f}}H^{\circ}_{\mathrm{m}}(\mathrm{CH_3})_2\mathrm{NH},\ \mathrm{g}\}],$$

which assumes the (P–N) bond enthalpies in the two phosphorus compounds are identical. Taking $\Delta_{\mathrm{f}}H^{\circ}_{\mathrm{m}}[\{(\mathrm{C_2H_5})_2\mathrm{N}\}_3\mathrm{P,g}] = -(225.6 \pm 10.5)\ \mathrm{kJ \cdot mol^{-1}}$ (Pilcher's calculation(11) of Fowell and Mortimer's result(12)), $\Delta_{\mathrm{f}}H^{\circ}_{\mathrm{m}}\{(\mathrm{C_2H_5})_2\mathrm{NH},\ \mathrm{g}\} = -(72.5 \pm 1.2)\ \mathrm{kJ \cdot mol^{-1}}$,(13) and $\Delta_{\mathrm{f}}H^{\circ}_{\mathrm{m}}\{(\mathrm{CH_3})_2\mathrm{NH},\ \mathrm{g}\} = -(18.6 \pm 0.8)\ \mathrm{kJ \cdot mol^{-1}}$:(13) $\Delta_{\mathrm{f}}H^{\circ}_{\mathrm{m}}[\{\mathrm{CH_3})_2\mathrm{N}\}_3\mathrm{P},\ \mathrm{g}] = -(63.9 \pm 11.3)\ \mathrm{kJ \cdot mol^{-1}}$, which does not differ significantly from the reported value of $-(71.3 \pm 2.4)\ \mathrm{kJ \cdot mol^{-1}}$.

A simple self-consistency test for the standard molar enthalpies of formation of a series of related compounds is to compare the magnitudes of the molar enthalpies of redistribution reactions:(11)

$$\tfrac{1}{3}\mathrm{PCl_3(g)} + \tfrac{2}{3}\{(\mathrm{CH_3})_2\mathrm{N}\}_3\mathrm{P(g)} = \{(\mathrm{CH_3})_2\mathrm{N}\}_2\mathrm{PCl(g)};$$

$$\Delta_{\mathrm{r}}H_{\mathrm{m}} = -(31.3 \pm 3.2)\ \mathrm{kJ \cdot mol^{-1}},$$

$$\tfrac{2}{3}\mathrm{PCl_3(g)} + \tfrac{1}{3}\{(\mathrm{CH_3})_2\mathrm{N}\}_3\mathrm{P(g)} = (\mathrm{CH_3})_2\mathrm{NPCl_2(g)};$$

$$\Delta_{\mathrm{r}}H_{\mathrm{m}} = -(30.9 \pm 3.1)\ \mathrm{kJ \cdot mol^{-1}}.$$

If the (P–N) and the (P–Cl) bond enthalpies were constant and transferable, then these redistribution molar enthalpies would be zero. That they are not reflects the enhanced stability of the substituted phosphines relative to the symmetrical analogues. Comparison of these redistribution molar enthalpies with corresponding but limited results for other P(III) compounds suggests that our results are satisfactorily consistent.(11) The mean bond-dissociation enthalpy for (P–N) in $\{(\mathrm{CH_3})_2\mathrm{N}\}_3\mathrm{P}$ is readily evaluated as $(274 \pm 8)\ \mathrm{kJ \cdot mol^{-1}}$, the main source of uncertainty arising from $\Delta_{\mathrm{f}}H^{\circ}_{\mathrm{m}}\{(\mathrm{CH_3})_2\mathrm{N},\ \mathrm{g}\} = (145 \pm 8)\ \mathrm{kJ \cdot mol^{-1}}$.(14) Mean bond-dissociation enthalpies for P(III) compounds have been tabulated by Pilcher(11) and range from $\langle D\rangle(\mathrm{P{-}F}) = 506\ \mathrm{kJ \cdot mol^{-1}}$ to $\langle D\rangle\{\mathrm{P{-}CH_2(CH_2)_2CH_3}\} = 194\ \mathrm{kJ \cdot mol^{-1}}$; our value for $\langle D\rangle(\mathrm{P{-}NMe_2}) = 274\ \mathrm{kJ \cdot mol^{-1}}$ lies at the lower end of this range and is similar in magnitude to $\langle D\rangle(\mathrm{P{-}Br})$ and $\langle D\rangle(\mathrm{P{-}CH_3})$.

Evaluation of bond enthalpies for the substituted phosphines requires the assumption of bond-enthalpy transferability of either $\langle D \rangle$(P–N) from $\{(CH_3)_2N\}_3P$ or of $\langle D \rangle$(P–Cl) from PCl_3; these approaches are equivalent. Assuming that $\langle D \rangle(\text{P–Cl}) = 322.2\ \text{kJ}\cdot\text{mol}^{-1}$ is transferable, then $\langle D \rangle$(P–N) in $(CH_3)_2NPCl_2$ and in $\{(CH_3)_2N\}_2PCl$ are $305\ \text{kJ}\cdot\text{mol}^{-1}$ and $290\ \text{kJ}\cdot\text{mol}^{-1}$, respectively. Ancillary quantities for these bond-enthalpy calcualtions are

$$\Delta_f H_m^\circ(\text{PCl}_3, \text{g}) = -(286.2 \pm 2.4)\ \text{kJ}\cdot\text{mol}^{-1},^{(11)}$$

$$\Delta_f H_m^\circ(\text{P}, \text{g}) = (316.5 \pm 1.0)\ \text{kJ}\cdot\text{mol}^{-1},^{(9)} \text{ and}$$

$$\Delta_f H_m^\circ(\text{Cl}, \text{g}) = (121.30 \pm 0.01)\ \text{kJ}\cdot\text{mol}^{-1}.^{(9)}$$

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