Vapor Pressures of Aluminum Tribromide and Aluminum Triiodide and Their Standard Sublimation Enthalpies

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Total vapor pressures of AlBr₃ and AlI₃ were measured by a torsion effusion apparatus. Their temperature dependences are expressed by the equations $\log(p/Pa) = (14.78 \pm 0.60) - (4700 \pm 200)/(T/K)$ from (301.0 to 351.0) K and $\log(p/Pa) = (15.52 \pm 0.30) - (5960 \pm 150)/(T/K)$ from (359.5 to 419.5) K for AlBr₃ and AlI₃, respectively. Practically, AlBr₃ vaporizes in a dimeric form, while AlI₃ vaporizes in monomeric and dimeric forms. Treating the vapor pressures of AlBr₃ by second- and third-law methods, the standard sublimation enthalpy $\Delta H^{\circ}(298 \text{ K}) = (90 \pm 4) \text{ kJ} \cdot \text{mol}^{-1}$ was derived. For AlI₃, treating the partial pressures of dimer-monomer equilibrium reported in the literature, by second- and third-law methods, the standard enthalpies $\Delta H^{\circ}(298 \text{ K}) = (110 \pm 4) \text{ kJ} \cdot \text{mol}^{-1}$ and $\Delta H^{\circ}(298 \text{ K}) = (119 \pm 4) \text{ kJ} \cdot \text{mol}^{-1}$ associated to the sublimation reactions of AlI₃ according to the AlI₃(s) \rightarrow AlI₃(g) and 2AlI₃(s) \rightarrow Al₂I₆(g), respectively, were selected.

Introduction

Apparently, the first reliable vapor pressure data on AlBr₃ were those measured above the molten compound by Fischer et al.¹ using a static method. These authors have shown that the dimeric form is practically the only species present in the vapor and established melting point and heat of fusion of this compound. From these data, the vapor pressures above the solid phase were evaluated and selected by Kelley.² Jonson et al.³ measured above the molten compound by an inverted capillary very high vapor pressures (from $1.20 \cdot 10^5$ Pa to $2.87 \cdot 10^6$ Pa). Dunne and Gregory⁴ by the Knudsen effusion method and Sulzmann⁵ by a precision capacitance micromanometer measured the vapor pressures above the solid compound. Recently, Rusin and Niesel'son⁶ studied at temperatures around the critical point, (763 ± 4) K, the complex equilibria involving monomer, dimer, and trimer forms in the gaseous phase.

Also, for AlI₃ the first vapor pressures were those measured above the liquid phase by Fischer et al.¹ using a static method. The authors have shown that, unlike other triiodides of IIIA group elements (GaI₃ and InI₃), the vapor consists of only monomer species in the vapor above AlI₃ which are present in both monomeric and dimeric forms. Gregory⁷ by absorbance measurements in the ultraviolet region shows that at low temperatures (around ambient) the vapor is virtually all Al₂I₆(g), but with increasing temperature, the dissociation process to the monomeric form becomes important. In a figure from this work⁷ are drawn the temperature dependence of the partial pressures of monomer and dimer species below the melting point, pressure values derived by the equilibrium constants of these forms taken from ref 1, and the measured total absorbance. Rusin,⁸ using the vapor pressure data from ref 1, recalculated the standard

Table 1. Torsion Total Vapor Pressures of AlBr₃

r	un 1	r	un 2	r	run 4		
T/K	$\log(p/Pa)$	T/K	$\log(p/Pa)$	T/K	log(p/Pa)		
309.0	-0.37	303.0	-0.75	314.0	-0.21		
315.5	-0.07	307.0	-0.45	316.0	-0.15		
318.5	0.03	310.0	-0.35	318.0	-0.05		
323.0	0.17	312.5	-0.27	320.0	0.03		
324.0	0.23	314.5	-0.15	323.0	0.23		
327.5	0.37	317.5	-0.05	324.0	0.25		
330.5	0.49	318.5	0.01	326.0	0.31		
333.5	0.64	320.5	0.09	328.0	0.43		
338.0	0.87	321.5	0.15	331.0	0.59		
343.0	1.10	324.0	0.25	335.0	0.76		
345.0	1.17	327.0	0.38	336.0	0.79		
348.0	1.29	333.0	0.66	338.0	0.90		
351.0	1.40	336.0	0.76	340.0	0.97		
				344.0	1.11		
r	un 6	r	un 8	run 9			
T/K	$\log(p/Pa)$	T/K	$\log(p/Pa)$	T/K	log(p/Pa)		
307.0	-0.47	301.0	-0.87	309.0	-0.46		
312.0	-0.25	306.0	-0.62	312.0	-0.31		
315.0	-0.14	312.0	-0.34	315.0	-0.17		
316.5	-0.05	317.0	-0.02	317.0	-0.08		
318.5	0.05	323.0	0.22	318.5	0.01		
321.0	0.13	325.0	0.32	320.0	0.09		
324.5	0.28	328.0	0.40	323.0	0.20		
327.0	0.45	330.0	0.51	326.0	0.33		
330.0	0.56	333.0	0.60				
332.5	0.67	336.0	0.75				
337.0	0.86	340.0	0.91				

enthalpy associated to the dimer-monomer equilibrium. As part of a systematic study on the vaporization of group III metal halides,⁹⁻¹¹ we have measured by a torsion method the total vapor pressures of aluminum tribromide and triiodide and derived their sublimation enthalpies treating the pressure data by the second- and third-law methods.

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Figure 1. Torsion total vapor pressures of AlBr₃. \bigcirc , run 1; \bigcirc , run 2; \triangle , run 4; \blacktriangle , run 6; \Box , run 8; \blacksquare , run 9.

Table 2. Temperature Dependence of the Total Vapor Pressures of $AlBr_3$ and AlI_3

		ΔT		$\log(p/\mathrm{Pa}) = A$	A - B/(T/K)
compound	run	K	points	A^a	B^{a}
AlBr ₃	1	309.0 to 351.0	13	14.70 ± 0.32	4675 ± 105
	2	303.0 to 336.0	13	14.26 ± 0.27	4537 ± 85
	4	314.0 to 344.0	14	15.48 ± 0.20	4936 ± 66
	6	307.0 to 337.0	11	14.64 ± 0.24	4648 ± 77
	8	301.0 to 340.0	11	14.68 ± 0.23	4677 ± 75
	9	309.0 to 326.0	8	14.83 ± 0.22	4725 ± 71
AlI ₃	1	365.0 to 417.5	14	15.05 ± 0.07	5757 ± 27
	2	361.0 to 417.0	15	15.31 ± 0.17	5880 ± 64
	3	361.0 to 416.0	14	15.71 ± 0.11	6041 ± 42
	5	368.0 to 414.5	15	15.77 ± 0.12	6062 ± 48
	6	359.5 to 419.5	15	15.74 ± 0.15	6047 ± 57
	8	367.5 to 416.0	14	15.63 ± 0.20	6000 ± 77
	9	364.0 to 415.0	15	15.44 ± 0.12	5928 ± 45

^a The quoted uncertainties are standard deviations.

Experimental and Results

Very pure samples of AlBr₃ and AlI₃ were supplied by Aldrich (both 99.999 % pure as stated by the same supplier). Both compounds are hygroscopic and easily hydrolyzed and oxidized so that the containers of the samples were opened and the cells filled with the samples in a efficient drybox in an argon atmosphere. Moreover, both effusion holes of the cell were closed employing small pins of naphthalene. These pins sublime quickly when the cell was put under vacuum. This procedure excludes the contact of the sample with air. The vapor pressures of AlBr₃ and AlI₃ were measured by the torsion assembly substantially described in the previous work.¹² A conventional graphite torsion cell, with the effusion holes having a nominal



Figure 2. Comparison of the total vapor pressures of AlBr₃. A, Fisher et al.;¹ B, Johnson et al.;² C, Dunne and Gregory;⁴ D, Sulzmann;⁵ E, this work.



Figure 3. Torsion total vapor pressures of AlI₃. \bigcirc , run 1; \bigcirc , run 2; \triangle , run 3; \blacktriangle , run 5; \square , run 6; \blacksquare , run 8; \diamondsuit , run 9.

diameter of 1 mm with knife edged orifices, was used in this study. The cell constant necessary to convert pressure data from experimental torsion angles was obtained vaporizing very pure benzoic acid and urea, the vapor pressures of which are well-known.^{13,14} The constant values obtained with both standards were found practically equal, and when it was checked in runs carried out during the study of the compounds, they were reproducible (within about 5 % of their average value). This uncertainty produces a very negligible shift in the intercepts of the final log *p* equations. In each vaporization run, the torsion angles, and then the vapor pressures, were measured randomly in both ascending and descending temperatures (though in the

Table 3.	Comparison of	of the	Temperature	Dependence	of the Tota	l Vapor	Pressures of	f AlBr ₃	and All

	1 1	1		U	5		
				ΔT	n° of points	$\log(p/Pa) = A$	A - B/(T/K)
compound	ref	method	equilibrium	K	points	Α	В
AlBr ₃	Fischer et al.1	static	liq-vap	391.5 to 522.7	18	10.33 ^a	2800 ^a
	Kelley ²	extrapolated from ref 1 data	sol-vap	320 to 355		15.262	4651
	Dunne and Gregory ⁴	Knudsen-effussion	sol-vap	273 to 310		14.31	4292
	Johnson et al. ³	static	liq-vap	536 to 761	18	9.68	2452
	Sulzmann ⁵	static	sol-vap	302.6 to 334.4	13	15.135	4611
	this work	torsion effusion	sol-vap	301.0 to 351.0	70	14.78 ± 0.60	4700 ± 200
AlI ₃	Fischer et al. ¹	static	liq-vap	493.8	20	10.76 ^a	3766 ^a
				645.2			
	this work	torsion effusion	sol-vap	359.5	102	15.52 ± 0.30	5960 ± 150
				419.5			

^a By us, calculated from the pressure data reported in Fisher's work.¹



Figure 4. Comparison of the partial pressures of AlI_3 (solid lines) and Al_2I_6 (dotted lines). A, Fischer et al.;¹ B, this work.

tables the data are reported as ascending), and at the beginning and the end of each experiment, the measurements were made at approximately equal temperatures.

AlBr₃. The vapor pressures of AlBr₃ are reported in Table 1 and Figure 1. Because the experimental operative temperatures of both AlBr₃ and AlI₃ are near ambient, where the vapor pressure values are high enough, then to thermostat the cell a long time was necessary with consequent sublimation of a large amount of sample. For this reason and considering that a condition for the correct use of the effusion method is an upper limit of the pressure in the cell, in each run a small temperature range was covered, and a limited number of points were determined. The experimental data of each run were treated by the linear least-squares method to represent the temperature dependence of the vapor pressures as a log p versus 1/Tequation. The obtained equations are reported in Table 2. Weighting the slopes and intercepts proportionally to the experimental points, the following equation representative of the total vapor pressures of AlBr₃ in the temperature range (301.0 to 351.0) K was selected

$$\log(p/Pa) = (14.78 \pm 0.60) - (4700 \pm 200)/(T/K)$$
(1)

where the associated uncertainties are estimated.

This equation is compared in Table 3 and Figure 2 with those found in the literature. From the slope of eq 1, the second-law enthalpy associated to the sublimation of AlBr₃, according the reaction

$$2AlBr_3(s) \rightarrow Al_2Br_6(g) \tag{2}$$

 $\Delta_{sub}H^{\circ}(326 \text{ K}) = (90 \pm 4) \text{ kJ} \cdot \text{mol}^{-1}$ was calculated. By thirdlaw treatment of the vapor pressures evaluated by eq 1 at two

Table 5. Torsion Total Vapor Pressures of AlI₃

r	un 1	r	un 2	r	run 3 run 5		un 5
<i>T</i> /K	log(p/Pa)	<i>T</i> /K	log(p/Pa)	<i>T</i> /K	log(p/Pa)	<i>T</i> /K	log(p/Pa)
365.0	-0.71	361.0	-1.01	361.0	-1.01	368.0	-0.71
368.0	-0.61	368.5	-0.61	366.5	-0.77	372.0	-0.53
374.5	-0.31	372.0	-0.53	371.5	-0.57	374.5	-0.44
377.0	-0.23	374.5	-0.41	376.0	-0.38	378.0	-0.27
380.5	-0.08	377.0	-0.23	378.5	-0.23	377.0	-0.31
385.0	0.10	381.0	-0.11	382.0	-0.08	380.0	-0.17
389.0	0.27	385.5	0.05	388.0	0.13	383.0	-0.03
393.0	0.39	390.0	0.23	393.5	0.33	387.0	0.13
397.5	0.56	394.0	0.41	398.0	0.54	391.0	0.23
402.0	0.73	397.0	0.51	401.0	0.64	396.5	0.47
406.0	0.88	400.0	0.62	405.0	0.78	399.5	0.61
409.0	0.97	404.5	0.75	408.0	0.89	403.5	0.75
413.0	1.11	409.5	0.92	412.0	1.07	408.0	0.89
417.5	1.26	413.0	1.06	416.0	1.19	411.0	1.02
		417.0	1.22			414.5	1.15

r	un 6	r	un 8	r	un 9	
T/K	log(p/Pa)	T/K	log(p/Pa)	T/K	log(p/Pa)	
359.5	-1.14	367.5	-0.71	364.0	-0.84	
365.0	-0.84	372.0	-0.53	368.0	-0.71	
369.5	-0.61	375.5	-0.36	372.0	-0.47	
372.5	-0.47	377.0	-0.23	376.0	-0.31	
376.0	-0.31	380.0	-0.15	378.5	-0.20	
380.5	-0.11	383.5	-0.06	381.0	-0.11	
385.0	0.01	388.0	0.13	385.5	0.05	
390.0	0.26	391.0	0.32	390.0	0.24	
395.0	0.40	395.0	0.45	393.5	0.37	
399.0	0.58	399.5	0.62	398.0	0.53	
403.0	0.72	403.5	0.74	402.0	0.70	
407.0	0.88	408.0	0.90	405.0	0.82	
411.0	1.01	412.0	1.07	408.5	0.94	
415.0	1.17	416.0	1.20	412.0	1.04	
419.5	1.34			415.0	1.16	

temperatures at the extremes of the experimental range, (300 and 350) K, two values of the standard enthalpy of the sublimation of AlBr₃ were calculated. The free energy functions (fef), $[G^{\circ}(T) - H^{\circ}(298 \text{ K})]/T$, necessary for these calculations were taken from the IVTANTHERMO database¹⁵ and by Pankratz.¹⁶ The fef of Al₂Br₆(g) reported by these references (see Table 4) are different because of use of two different values of the absolute standard entropy, $S^{\circ}(298 \text{ K}) = (537 \text{ and } 547)$ $J \cdot K^{-1} \cdot mol^{-1}$, for refs 15 and 16, respectively. The third-law $\Delta_{\rm sub}H^{\circ}(298 \text{ K})$ values so obtained, and those recalculated from Dunne's⁴ and Sulzmann's⁵ vapor pressures evaluated at (300 and 350) K from the log p versus 1/T equations reported in their works, are reported in Table 4. In Table 4 are also reported the second-law sublimation enthalpy values, all reported at 298 K using an enthalpic increment of $1 \text{ kJ} \cdot \text{mol}^{-1}$.^{15,3} Both our thirdlaw enthalpies (86.5 and 89.9) kJ·mol⁻¹ obtained from the fef of refs 15 and 16, respectively, are higher than those reported by the other authors. It is difficult to choose between our two enthalpy values, but the lack of a temperature trend in the

Table 4. Standard Sublimation Enthalpy of AlBr₃ According to the Reaction: $2AlBr_3 \rightarrow Al_2Br_6(g)$

			Dunne and Gregory ⁴			S	Sulzmann ⁵			this work		
	$\Delta[(G^{\circ}(T) - I)]$	H°(298 K)/T]	third-la	third-law $\Delta H^{\circ}(298 \text{ K})$			third-law $\Delta H^{\circ}(298 \text{ K})$			third-law $\Delta H^{\circ}(298 \text{ K})$		
Т	$J \cdot K^{-1}$	mol ⁻¹	p	kJ∙n	nol^{-1}	р	p kJ·mol ⁻¹		р	kJ∙mol ^{−1}		
K	from ref 15	from ref 16	Pa	а	b	Pa	а	b	Ра	а	b	
300	-176.54	-186.62	1.008	81.7	84.7	0.5821	83.1	86.1	0.1254	86.9	89.9	
350	-176.20	-186.68	111.5	81.4	85.2	91.35	82.0	85.7	21.78	86.2	89.9	
			average	81.6	84.9	average	82.5	85.9	average	86.5	89.9	
			second-law $\Delta H^{\circ}(298 \text{ K}) =$		second-law $\Delta H^{\circ}(298 \text{ K}) =$			second-law $\Delta H^{\circ}(298 \text{ K}) =$				
			83.2 kJ·mol ⁻¹		89.3 kJ \cdot mol ⁻¹			$91 \pm 4 \text{ KJ} \cdot \text{mol}^{-1}$				

^a Values obtained by using the fef from ref 15. ^b Values obtained by using the fef from ref 16.

Table 6. Third-Law Standard Sublimation Enthalpies Associated to the Sublimation of Solid AlI₃ According to Reactions 6 and 7 (See Text)

		rea	action 5: AlI ₃ (s)	$\rightarrow AlI_3(g)$	reaction 6: $2AII_3(s) \rightarrow Al_2I_6(g)$					
Т	p_{All_3}	Δfef^a	$\Delta H^{\circ}(298 \text{ K})^a$	Δfef^b	$\Delta H^{\circ}(298 \text{ K})^b$	$p_{\mathrm{Al}_2\mathrm{I}_6}$	Δfef^a	$\Delta H^{\circ}(298 \text{ K})^a$	Δfef^b	$\Delta H^{\circ}(298 \text{ K})^b$
Κ	Pa	$J \cdot K^{-1} \cdot mol^{-1}$	kJ∙mol ^{−1}	$J \cdot K^{-1} \cdot mol^{-1}$	$kJ \cdot mol^{-1}$	Pa	$\overline{J \cdot K^{-1} \cdot mol^{-1}}$	kJ∙mol ^{−1}	$\overline{J \cdot K^{-1} \cdot mol^{-1}}$	kJ∙mol ⁻¹
360	0.0323	183.3	110.8	211.0	120.7	0.0603	209.0	118.2	72.7	141.1
420	5.31	182.5	111.0	213.2	124.0	16.2	207.9	117.8	277.6	147.1
			110.9		122.3			118.0		144.1

^a Values obtained by using the fef from ref 15. ^b Values obtained by using the fef from ref 16.

sublimation enthalpies obtained from the fef of ref 16 and the better agreement with the second-law enthalpy value (91 \pm 4) kJ·mol⁻¹, although considering the small experimental temperature range this value is less reliable than that obtained from the third-law, lead one to propose as the more reliable value of the standard sublimation enthalpy of AlBr₃ 90 kJ·mol⁻¹, with an overestimated uncertainty of 4 kJ·mol⁻¹.

*AlI*₃. The total vapor pressures of solid AlI₃ are reported in Table 5 and Figure 3. By least-squares treatment of the data measured in each run, the temperature dependence of the total vapor pressures was represented as a log *p* versus 1/T equation. The obtained equations are reported in Table 2. Weighting slopes and intercepts of these equations proportionally to the experimental points, an equation representative of the total vapor pressure of solid AlI₃ in the temperature range (359.5 to 419.5) K was selected

$$\log(p/Pa) = (15.52 \pm 0.30) - (5960 \pm 150)/(T/K)$$
(3)

where the associated uncertainties have been estimated.

Considering that in the vapor above the solid phase both monomeric and dimeric forms are present, the temperature dependence of their partial pressures was derived from eq 3 and from the temperature dependence of the constant of the dimer-monomer equilibrium, $\log[(p_{AII_3}^2/p_{AI_2I_3})/Pa] = 5045 - 12.253/(T/K)$, the equation obtained from that reported by Fischer et al.¹ above the molten compound and the heat of fusion (15.9 kJ·mol⁻¹) selected by ref 15. The temperature dependence of the partial pressures of AlI₃(g) and Al₂I₆(g) is expressed by the equations

$$\log(P_{AIL}p/Pa) = 14.02 - 5582/(T/K)$$
(4)

$$\log(P_{AI_2I_6}p/Pa) = 15.78 - 6119/(T/K)$$
(5)

The uncertainties associated with the slopes and intercepts of these equations are difficult to evaluate but can be considered comparable with those estimated in eq 3. In Figure 4, these equations are drawn for comparison with those reported by Fischer et al.¹ above the liquid phase. The second-law enthalpies of solid AlI₃ according to the processes

$$All_3(s) \rightarrow All_3(g)$$
 (6)

$$2\text{AlI}_3(s) \rightarrow \text{Al}_2 I_6(g) \tag{7}$$

were calculated at the average experimental temperature, $\Delta_{sub}H^{\circ}(389.5 \text{ K}) = 107 \text{ kJ}\cdot\text{mol}^{-1}$ and $117 \text{ kJ}\cdot\text{mol}^{-1}$ for reactions, 7 respectively. These values are corrected to 298 K by using the enthalpic increments for solid and gaseous aluminum iodides selected by ref 15, $\Delta_{sub}H^{\circ}(298 \text{ K}) = 109$ kJ·mol⁻¹ and 120 kJ·mol⁻¹ for reactions 6 and 7, respectively, with an uncertainty that should not exceed 6 kJ·mol⁻¹ for both reactions. These standard enthalpies were also calculated by third-law treatment of the partial pressures of AlI₃(g) and Al₂I₆(g) obtained at the extreme experimental temperatures (360 and 420) K by eqs 4 and 5. The fef necessary for the calculations were taken from ref 15 and 16. The fef of solid AlI₃ are different because of the use of a different standard entropy, S°(298 K) = 190 $J \cdot K^{-1} \cdot mol^{-1}$ from ref 15 (taken from JANAF¹⁷) and 158.9 $J \cdot K^{-1} \cdot mol^{-1}$ by ref 16 (taken from Volgman¹⁸). The obtained enthalpy values are reported in Table 6. The thirdlaw enthalpies of both sublimation reactions 6 and 7 obtained by using the Δ fef from ref 15 are more reliable than those obtained by using the Δ fef from ref 16 because of negligible temperature trends and are in better agreement with the secondlaw results. On this basis, we propose as partial standard sublimation enthalpies of AlI₃ according to reactions 6 and 7 the values 110 kJ·mol⁻¹ and 119 kJ·mol⁻¹, respectively, with an overestimated uncertainty for both reactions of 4 kJ \cdot mol⁻¹, with a recommended standard entropy of solid AlI₃ of 190 $J \cdot K^{-1} \cdot mol^{-1}$ proposed by ref 15.

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