Vaporization Study of SmI_3 and SmI_2

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The decomposition and vaporization processes of samarium triiodide were studied. The enthalpy changes associated with the decomposition reaction, $SmI_3(s) \rightarrow SmI_2(l) + {}^1/_2I_2(g)$, $\Delta H°(298\ K) = (117 \pm 10)\ kJ\cdot mol^{-1}$, and the sublimation process, $SmI_3(s) \rightarrow SmI_3(g)$, $\Delta_{sub}H°(298\ K) = 276\ kJ\cdot mol^{-1}$, were determined from the temperature dependence of the equilibrium vapor pressures measured extensively in an appropriate temperature interval. The congruent vaporization of samarium diiodide was also studied, and the value for its standard sublimation enthalpy was selected to be $\Delta_{sub}H°(298\ K) = (292 \pm 5)\ kJ\cdot mol^{-1}$.

Introduction

The vaporization of SmI_2 was studied in a previous work.[1] The standard sublimation enthalpy of this compound, determined by the second-law treatment of the vapor pressure data, $\Delta_{sub}H°(298\ K) = (272 \pm 10)\ kJ\cdot mol^{-1}$, is lower than an old value estimated by Feber[2] (293 $kJ\cdot mol^{-1}$) and that selected by Hirayama et al.,[3] (291 ± 8) $kJ\cdot mol^{-1}$. Gorokhov et al.,[4] in a mass spectrometric study of SmI_3 that decomposes on heating to SmI_2, measured the vaporization enthalpy of SmI_2 in the molten state at 1150 K, and the obtained value, $\Delta_{vap}H°(1150\ K) = (250 \pm 7)\ kJ\cdot mol^{-1}$, is in agreement with the results of Knudsen effusion [$\Delta_{vap}H°(1082\ K) = (256 \pm 5)\ kJ\cdot mol^{-1}$] and mass spectrometric measurements [$\Delta_{vap}H°(1061\ K) = (268 \pm 13)\ kJ\cdot mol^{-1}$] by Hirayama et al.[3] It is interesting to compare the above standard vaporization enthalpy values of SmI_2 with those of other two rare earth diiodides—EuI_2,[5] (309 ± 13) $kJ\cdot mol^{-1}$, and YbI_2, (302±6) $kJ\cdot mol^{-1}$—measured by us in a recent work.[6] Considering these results, we find that the low value for the sublimation enthalpy of SmI_2 that we proposed in our previous work[1] did not appear to be correct. Therefore, in the present study we reinvestigate the vaporization of SmI_2 to obtain a new value for its sublimation enthalpy. A study of the vaporization of SmI_3 was also carried out. It is known[4,7,8] that this compound readily decomposes on heating, giving up iodine with the formation of SmI_2. During the decomposition of SmI_3, in addition to a large amount of I_2 the presence of a small amount of $SmI_3(g)$ was also observed in the vapor by Gorokhov et al.[4]

Experiment and Results

Samples of SmI_3 and SmI_2 with a nominal purity of 99.8 % were supplied by Aldrich.

The vapor pressures above these compounds were measured by the torsion technique[9] using the same assembly described in a previous work.[10] Three conventional torsion cells machined from different materials—graphite (cell E), quartz (cell F), and pyrophyllite (cell A) (having effusion holes 0.5, 1.2, and 1.5 mm in diameter, respectively)—were used to check the effects of the possible interaction of the vapor phase with the cell material. The vapor pressure above SmI_2 was detected at temperatures where the compound was in the molten state. The samples were loaded in cells inserted into a small quartz wire nest to increase the vaporization surface and to minimize any “creeping-out” effect. To prevent contact with the atmosphere and hydration of the samples, the cells were loaded in a drybox. The cell constants of the cells used, which are necessary to convert the experimental torsion angles to pressure values, were determined by vaporizing standard substances with well-known vapor pressures[11] (cadmium and lead in this work), and their values were checked during the study of each compound. The uncertainties associated with the calibration constants should affect the measured pressure values by no more than 5 % of the reported values, and this produces in the logarithm of the absolute pressure values a displacement of about ±0.02. The error in the temperature measurements, carried out by using a calibrated Pt−Pt(Rh 10 %) thermocouple inserted into a cell placed beneath the torsion apparatus, should not exceed ±2 K in the experimental temperature ranges covered in this study.

The overall reliability of the torsion apparatus was checked during the calibration runs by measuring the second-law sublimation enthalpy of pure lead from the slope of a plot of the logarithm of the torsion angles versus $^1/_T$. All of the enthalpy values so determined using the different cells agree, within their standard deviations, with those selected by Hultgren et al.[11]

A. SmI_2. In Table 1 and Figure 1, the results obtained for this compound are summarized. In Figure 1, we also report the log *p* versus $^1/_T$ lines that we obtained in the previous work[1] and that selected by Hirayama et al.[3] Slopes and intercepts of the log *p* versus $^1/_T$ equations evaluated by treating the experimental data of each run by least squares are reported in Table 2 along with the results from our previous work.[1] The vapor pressures measured in the single run (E3) with the graphite cell are in better agreement with those measured in our previous work[1] (Table 2) and in particular with those obtained by using graphite cell C having smaller effusion orifices (runs C1 and C2). The other vapor-pressure values measured in the present work with quartz pyrophyllite cells are in better agreement

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Table 1. Torsion Vapor Pressure Measured over Liquid SmI_2

run E3		run F2		run F4	
T/K	−log(*p*/kPa)	*T*/K	−log(*p*/kPa)	*T*/K	−log(*p*/kPa)
1080	3.40	1076	3.57	1076	3.62
1088	3.32	1093	3.37	1089	3.46
1102	3.13	1108	3.19	1100	3.34
1121	2.93	1129	2.97	1110	3.21
1128	2.86	1145	2.80	1120	3.10
1140	2.75	1160	2.64	1129	3.02
1159	2.58	1169	2.56	1139	2.91
1171	2.46	1177	2.48	1150	2.80
1177	2.42	1186	2.40	1158	2.70
1183	2.35			1168	2.62
				1177	2.54
				1186	2.43

run A4		run A5		run A7	
T/K	−log(*p*/kPa)	*T*/K	−log(*p*/kPa)	*T*/K	−log(*p*/kPa)
1048	3.87	1051	3.80	1065	3.73
1068	3.62	1064	3.67	1072	3.67
1082	3.49	1074	3.57	1092	3.46
1092	3.37	1085	3.46	1100	3.34
1103	3.25	1096	3.32	1108	3.29
1114	3.11	1104	3.23	1118	3.16
1124	3.03	1114	3.11	1125	3.07
1136	2.87	1124	3.02	1133	2.98
1145	2.78	1133	2.91	1142	2.89
1154	2.69	1142	2.81	1151	2.80
1155	2.67	1152	2.70	1158	2.72
1164	2.58	1161	2.62	1162	2.68
1173	2.51	1169	2.54	1171	2.60
1181	2.43	1179	2.45	1179	2.53
1186	2.39	1188	2.37	1186	2.49
				1189	2.47

with Hirayama's[3] data [log(*p*/kPa) = (8.93 ± 0.26) − (13 367 ± 275)/(*T*/K)].

It is interesting that the slopes of the equations obtained by using quartz and pyrophyllite cells are slightly greater than those obtained by graphite cells. Probably because of the absorption effects caused by the porosity of the cell material, in the experiments carried out with graphite cells the activity of the molten sample (and therefore the pressure data) shows a very small decrease with time in the vaporization rate. Giving therefore more weight to the five equations obtained with the quartz and pyrophyllite cells, we selected the following equation, weighting the corresponding slope and intercept of the single run proportionally to the number of experimental points

$$\log(p/\text{kPa}) = (8.92 \pm 0.10) - (13\,440 \pm 100)/(T/\text{K}) \quad (1)$$

where the associated errors were estimated. This equation is practically equal to that selected by Hirayama.[3]

B. SmI_3. Mass Spectra of SmI_3. The mass spectra of the vapor over SmI_3 were analyzed by a high-temperature Nuclide HT7 mass spectrometer. In the initial stage of heating the sample, I^+ and I_2^+ ion currents derived from the vaporization of a small amount of iodine present as an impurity in the sample were observed in the spectrum. Upon increasing the temperature, at about 700 K the sample was purified. (The small intensities of the I^+ and I_2^+ ions present in the spectrum were not shutterable.) At about 780 K, shutterable I^+ and I_2^+ ions due to the beginning of the decomposition of SmI_3 were observed in the mass spectrum. As heating progressed, in addition to high I^+ and I_2^+ ion intensities, very small ion intensities of Sm^+, SmI^+, SmI_2^+, and SmI_3^+ ions were also detected, with the SmI^+ ion being the most abundant. Owing to their small intensities, the appearance potentials of these ions, measured by the linear extrapolation method, could not be evaluated well, but their approximate values (measured to ±1 eV) demonstrate that all of these ions were derived from the fragmentation of SmI_3 molecules and this showed a partial congruent evaporation of SmI_3. During this first stage of vaporization, the ion intensities were reproducible for ascending and descending temperature for a limited vaporization time. The temperature dependence of the intensity of the most abundant ion, SmI^+, measured in this stage of the experiment is shown in Figure 2. Upon further vaporization, all ion intensities decreased at constant temperature, as was also observed by Gorokhov et al.[4] After heating to higher temperature (~950 K), the ions containing samarium were not detectable, and the iodine ions present in the mass spectra were not shutterable. Upon further heating to higher temperatures (~1050 K) when the vaporization of pure $SmI_2(s)$ to $SmI_2(g)$ started to be detectable, Sm^+, SmI^+, and SmI_2^+ ions were again observed in the mass spectrum with relative intensities (about 60:100:20, respectively) and appearance potentials (12, 11, and 9 eV, respectively, all ±0.5 eV) similar to those determined by Hirayama et al.[4] and Gorokhov et al.[5] The presence of the $Sm_2I_3^+$ ion was also observed in the spectrum, but its intensity was negligible compared to that of SmI^+; therefore, the concentration of the dimer precursor, $Sm_2I_4(g)$, was considered to be negligible (<0.1 % of the vapor pressure) compared to that of $SmI_2(g)$. In conclusion, the mass spectra of the vapors issuing from SmI_3 showed that, after the vaporization of a small iodine impurity in the first step, at about 800 K the principal decomposition process, $SmI_3(s) \rightarrow SmI_2(l) + {}^1\!/_2 I_2(g)$, and the congruent sublimation process, $SmI_3(s) \rightarrow SmI_3(g)$, occur simultaneously. As the experiment proceeds, $SmI_2(l)$ [mp (799 ± 3) K[12]], formed in the SmI_3 decomposition, reduces its activity so that all ion intensities decrease with time at constant temperature. After all SmI_3 was decomposed to SmI_2 at higher temperatures, the vaporization of SmI_2 was observed.

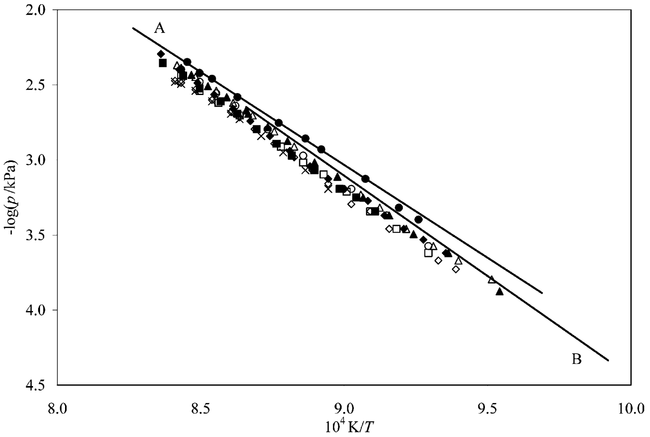

Figure 1. Torsion vapor pressures above SmI_2: ●, run E3; ○, run F2; ■, run F3; □, run F4; ▲, run A4; △, run A5; ◆, run A6; ◇, run A7; ×, run A9; A, ref 1; B, ref 3.

Torsion Vapor Pressures above SmI_3. The vapor pressures above SmI_3 during its heating were measured by the torsion method. At about 800 K, it was observed that SmI_3 decomposed to SmI_2 and that the vapor was mainly $I_2(g)$, with the monomeric form I(g) and $SmI_3(g)$ being negligible as mass-spectrometrically observed. In Table 3 and Figure 3, the $I_2(g)$ pressures measured in three runs using the quartz and pyrophyllite cells are reported. In Table 3, we also report the log *p* versus $^1\!/_T$ equations obtained by a least-squares treatment of the data of each run. From these equations, the following one was selected

Table 2. Temperature Dependence of the Vapor Pressures over Liquid SmI_2[a]

					ΔT	$\log(p/\text{kPa}) = A - B/(T/\text{K})$	
source	sample	cell	run	no. of points	K	A	B
our previous work[1]	SmI_2	graphite[b]	A1	13	1032−1179	8.24 ± 0.10	12 530 ± 106
	SmI_2	graphite[c]	B1	11	1047−1185	7.95 ± 0.12	12 214 ± 134
	SmI_2	graphite[c]	B2	9	1060−1128	8.01 ± 0.20	12 271 ± 219
	SmI_2	graphite[c]	B3	13	1042−1193	7.98 ± 0.10	12 234 ± 116
	SmI_2	graphite[c]	B4	12	1063−1167	7.98 ± 0.10	12 214 ± 116
	SmI_2	graphite[d]	C1	12	1118−1189	8.59 ± 0.18	12 869 ± 212
	SmI_2	graphite[d]	C2	11	1143−1210	8.45 ±0.08	12 720 ±97
this work	SmI_2	graphite	E3	10	1080−1183	8.50 ± 0.17	12 831 ± 191
	SmI_2	quartz	F2	9	1076−1186	9.13 ± 0.06	13 661 ± 73
	SmI_3	quartz	F3	12	1098−1195	8.99 ± 0.09	13 550 ± 104
	SmI_2	quartz	F4	12	1076−1186	9.04 ± 0.09	13 609 ± 106
	SmI_2	pyrophyllite	A4	15	1048−1186	9.02 ± 0.09	13 525 ±100
	SmI_2	pyrophyllite	A5	15	1051−1188	8.89 ± 0.10	13 373 ± 117
	SmI_3	pyrophyllite	A6	16	1069−1196	8.87 ± 0.13	13 389 ± 142
	SmI_2	pyrophyllite	A7	16	1065−1189	8.79 ± 0.14	13 348 ± 161
	SmI_3	pyrophyllite	A9	10	1118−1189	8.87 ± 0.32	13 455 ± 367

[a] Errors associated with slopes and intercepts are standard deviations. [b] Cells having effusion holes 2 mm in diameter. [c] Cells having effusion holes 1 mm in diameter. [d] Cells having effusion holes 0.6 mm in diameter.

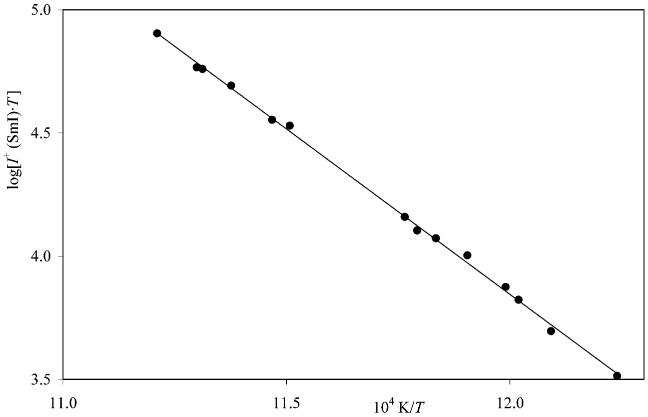

Figure 2. Temperature dependence of the SmI^+ ion above SmI_3(s).

where the errors were estimated. Table 3 includes only data determined in the first step of the decomposition process of SmI_3, when the small amount of molten SmI_2 formed and stratified on the surface of the sample does not appreciably influence the activity of SmI_3, and the I_2(g) pressure values were reproducible. As vaporization proceeded, at a constant temperature, the vapor pressure decreased continuously (presumably because of an increases in the amount of liquid SmI_2 covering the SmI_3 sample, reducing its activity). During this stage, all of the SmI_3 decomposed to SmI_2 so that, after further heating of the sample, the measured pressures were those in equilibrium over practically pure SmI_2. These pressures are also reported in Table 3. Considering the Sm_2I_4 partial pressure to be negligible and in any case minor or at the most comparable to the uncertainties associated with the torsion pressure values, the vapor pressures measured are considered to be SmI_2(g) partial pressures. The results, drawn in Figure 1 together with those measured above pure SmI_2 samples, are in good agreement. The three derived log p versus $^1/_T$ equations reported in Table 3 are compared in Table 2 with those found above the pure SmI_2 and those reported in the previous work.[4]

On this basis, considering all of the results obtained above SmI_2 in the present work to be in internal agreement, we propose as a final equation representative of its vapor pressure the selected eq 1.

Table 3. Torsion Vapor Pressures Measured during the Decomposition of SmI_3(s) and above SmI_2(l)[a]

run F3[b]		run A6[c]		run A9[d]	
T/K	$-\log(p/\text{kPa})$	T/K	$-\log(p/\text{kPa})$	T/K	$-\log(p/\text{kPa})$
799	3.69	789	3.77	801	3.69
807	3.54	797	3.63	808	3.60
819	3.30	805	3.47	815	3.47
824	3.19	814	3.30	823	3.33
833	3.06	823	3.15	832	3.17
840	2.95	833	3.00	840	3.01
849	2.83	843	2.84	849	2.87
856	2.72	852	2.67	857	2.77
863	2.63			867	2.63
868	2.56	1069	3.62	876	2.51
		1078	3.53		
1098	3.34	1086	3.46	1118	3.19
1106	3.25	1094	3.37	1128	3.07
1113	3.19	1101	3.27	1138	2.95
1124	3.07	1111	3.19	1148	2.84
1134	2.97	1118	3.13	1158	2.73
1141	2.89	1126	3.04	1162	2.69
1150	2.80	1135	2.94	1171	2.61
1159	2.69	1144	2.84	1179	2.54
1167	2.61	1153	2.74	1186	2.49
1177	2.52	1161	2.66	1189	2.48
1185	2.44	1170	2.56		
1195	2.35	1178	2.49		
		1187	2.40		
		1196	2.29		

[a] All of the quoted errors are standard deviations. [b] run F3: $\log(pI_2(g)/\text{kPa}) = (10.48 \pm 0.30) - (11\,295 \pm 249)/(T/\text{K})$; $\log(pSmI_2(g)/\text{kPa}) = (8.99 \pm 0.09) - (13\,550 \pm 104)/(T/\text{K})$. [c] run A6: $\log(pI_2(g)/\text{kPa}) = (10.96 \pm 0.16) - (11\,622 \pm 133)/(T/\text{K})$; $\log(pSmI_2(g)/\text{kPa}) = (8.87 \pm 0.13) - (13\,389 \pm 142)/(T/\text{K})$. [d] run A9: $\log(pI_2(g)/\text{kPa}) = (10.48 \pm 0.22) - (11\,361 \pm 187)/(T/\text{K})$; $\log(pSmI_2(g)/\text{kPa}) = (8.87 \pm 0.32) - (13\,455 \pm 367)/(T/\text{K})$.

$$\log(p/\text{kPa}) = (10.6 \pm 0.4) - \left(\frac{11\,400 \pm 300}{T/\text{K}}\right) \qquad (2)$$

Discussion

A. SmI_3. The decomposition process

$$SmI_3(s) \rightarrow SmI_2(l) + {}^1/_2 I_2(g) \qquad (3)$$

was investigated by measuring the pressure of iodine produced during this process. From the slope of the selected eq 2, the second-law enthalpy associated with this process,

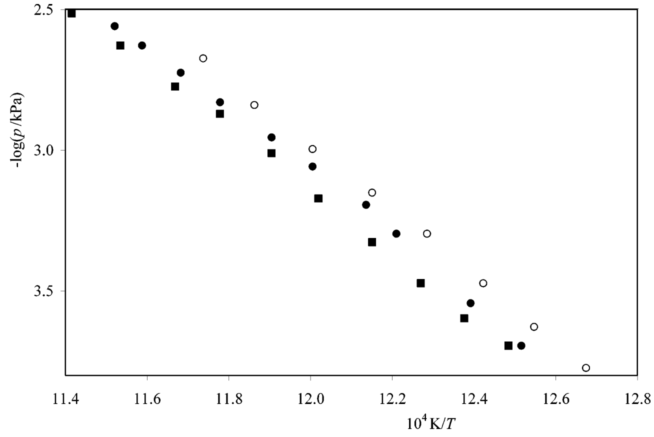

Figure 3. Temperature dependence of the iodine pressure during the decomposition of SmI_3 in SmI_2: ●, run F3; ○, run A6; ■, run A9.

Table 4. Third-Law Enthalpy of the Decomposition Process: $SmI_3(s) \rightarrow SmI_2(l) + {}^1/_2I_2(g)$

		$-[G°(T) - H°(298\ K)]/T$/J·K^{-1}·mol^{-1}			
T/K	−log(*p*/kPa)[a]	$SmI_3(s)$[b]	$SmI_2(l)$[c]	$I_2(g)$[d]	Δ*H*°(298 K)/ kJ·mol^{-1}
800	3.65	230	191	274	122
900	2.07	240	202	276	125

[a] Calculated from eq 2. [b] Calculated from ref 13. [c] Calculated from the thermodynamic data reported by Hirayama et al.[3] [d] Calculated from ref 11.

Δ*H*°(830 K) = (109 ± 3) kJ·mol^{-1}, was evaluated. This value was extrapolated to 298 K, Δ*H*°(298 K) = (110 ± 5) kJ·mol^{-1}, using as enthalpic increments, *H*°(830 K) − *H*°(298 K), the values 19.9, 61, and 50 kJ·mol^{-1} for $I_2(g)$,[11] $SmI_3(s)$,[13] and $SmI_2(l)$, respectively. The value for $SmI_2(l)$ was derived from the results reported in Hirayama's work.[3] The estimated error in Δ*H*°(298 K) takes into account the uncertainty introduced with extrapolation to 298 K. A third-law evaluation of the standard enthalpy of process 3 was made by using the free-energy function $[G°(T) - H°(298\ K)]/T$ taken from same sources as the enthalpic increments. In Table 4, we reported the results obtained at 800 and 900 K, the extreme temperatures of the interval over which the iodine pressures were measured. Considering the uncertainties in the evaluation of the thermodynamic functions, the results obtained are not in conflict with the second-law value. Giving equal weight to the second- and third-law results, we propose as the standard enthalpy of the decomposition process of SmI_3 the value Δ*H*°(298 K) = 117 kJ·mol^{-1} with an estimated error of 10 kJ·mol^{-1}, which reflects the large uncertainty in the thermal functions used.

The presence of small amounts of $SmI_3(g)$ in the vapor during the decomposition process of SmI_3 also allows us to study the sublimation process:

$$SmI_3(s) \rightarrow SmI_3(g) \tag{4}$$

From the slope of the mass-spectrometric data reported in Figure 2 and represented by the equation $\log[I^+(SmI)T] = 19.7 - 13\,480(K/T)$, the second-law sublimation enthalpy of this process was evaluated to be $\Delta_{sub}H°(850\ K) = 258$ kJ·mol^{-1}. It is difficult to estimate and associate a reliable error with this value, but we believe that it should not exceed ±10 kJ·mol^{-1}. This enthalpy, reduced to 298 K by using an estimated enthalpic increment of 18 kJ·mol^{-1} [average of corresponding values[14] for LaI_3 (18 kJ·mol^{-1}), CeI_3 (19 kJ·mol^{-1}), PrI_3 (20 kJ·mol^{-1}), GdI_3 (15 kJ·mol^{-1}), and TbI_3 (17 kJ·mol^{-1})], $\Delta_{sub}H°(298\ K) = 276$ kJ·mol^{-1}, is comparable with the values determined for other lanthanide triiodides[1] (Figure 4). It is difficult to evaluate accurately the $SmI_3(g)$ partial pressure from the present mass-spectrometric measurements because of various uncertainties (small ion intensity, ion fragmentation contribution, evaluation of molecular ionization cross section, multiplex gain, etc.). A SmI_3 partial pressure value was derived at 850 K by a third-law treatment of the measured standard sublimation enthalpy using as the difference in the free energy functions between solid and gaseous SmI_3 the value $\Delta[G°(T) - H°(298\ K)]/T = -206$ J·K^{-1}·mol^{-1}, obtained from the data proposed by Gorokhov et al.[11] The value obtained, 6×10^{-5} kPa, is comparable with those of other lanthanide triiodides ranging from 2×10^{-5} kPa (TmI_3)[15] to 3×10^{-6} kPa (LaI_3)[16] at the same temperature.

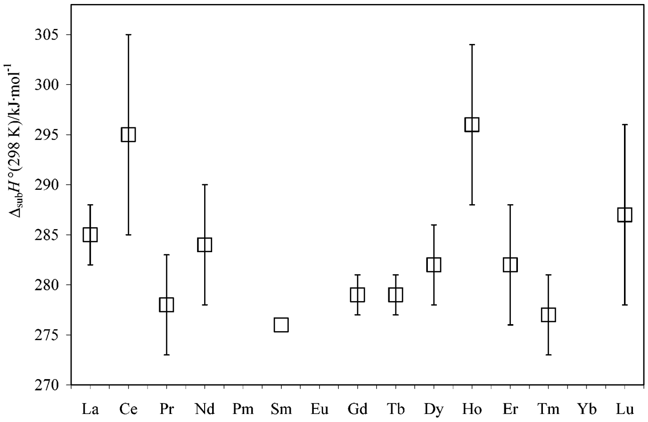

Figure 4. Standard sublimation enthalpies of lanthanide iodides.

The vapor pressures measured after SmI_3 was completely decomposed to SmI_2 were found to be essentially equal to those measured above pure SmI_2. The pressures measured in this work and represented by eq 1 are in better agreement with those proposed by Hirayama et al.[3] than those measured in our previous work.[1] From the slope of eq 1, the second-law vaporization enthalpy of SmI_2 was determined to be $\Delta_{vap}H°(1122\ K) = (257 \pm 3)$ kJ·mol^{-1}. This value is in close agreement with those obtained at comparable temperatures by Hirayama et al.[3] [Knudsen effusion $\Delta_{vap}H°(1082\ K) = (256 \pm 5)$ kJ·mol^{-1} and mass-spectrometric $\Delta_{vap}H°(1061\ K) = (268 \pm 13)$ kJ·mol^{-1}] and by Gorokhov et al.[4] [$\Delta_{vap}H°(1150\ K) = (249 \pm 7)$ kJ·mol^{-1}] and indicates that the sublimation enthalpy for SmI_2 that we reported previously,[1] $\Delta_{vap}H°(1121\ K) = (237 \pm 4)$ kJ·mol^{-1}, should be considered to be a lower limit. On this basis, utilizing the enthalpy increment derived from Hirayama's work,[3] $H°(1082\ K) - H°(298\ K) = 35$ kJ·mol^{-1}, we propose as the final value for the standard sublimation enthalpy of SmI_2 the value $\Delta_{sub}H°(298\ K) = (292 \pm 5)$ kJ·mol^{-1}, which comparable with those of EuI_2[5] and YbI_2,[6] (309 ± 13) kJ·mol^{-1} and (302 ± 6) kJ·mol^{-1}, respectively. These are apparently the only two lanthanide diiodides for which $\Delta_{sub}H°(298\ K)$ is reported in the literature.

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