Thermochemistry of Gaseous Species in the Dy−Br and Dy−I Systems

D. L. Hildenbrand* and K. H. Lau
SRI International, Menlo Park, California 94025

J. W. Baglio and C. W. Struck
Osram Sylvania, Inc., Beverly, Massachusetts 01915

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The gaseous monohalide, dihalide, and trihalide species in the Dy−Br and Dy−I systems were generated by the reactions of $Br_2(g)$ and $I_2(g)$ with Dy(s) in a heated effusion cell reactor, and equilibria among these species were studied in the range of 1300−1500 K by means of mass spectrometric monitoring. Reaction enthalpies were evaluated from third-law analysis of the equilibrium data and were used to derive the individual bond dissociation energies (BDE) in the species and related thermochemical properties. The analysis yielded the selected BDE values at 298 K: $D(\mathrm{Dy{-}Br}) = 81.1 \pm 2.5$, $D(\mathrm{BrDy{-}Br}) = 96.1 \pm 3.2$, and $D(\mathrm{Br_2Dy{-}Br}) = 106.0 \pm 3.8$ kcal mol^{-1}; and $D(\mathrm{Dy{-}I}) = 64.3 \pm 2$, $D(\mathrm{IDy{-}I}) = 80.1 \pm 2.8$, and $D(\mathrm{I_2Dy{-}I}) = 82.8 \pm 3.5$ kcal mol^{-1}. Bond energy sums in the two trihalides are in reasonable agreement with atomization enthalpies derived from sublimation data. D_0° values of DyBr and DyI are compared with recent estimates based on ligand field theory.

Introduction

Although thermochemical data have been reported for some gaseous monofluorides, difluorides, and trifluorides of the fourteen lanthanide (Ln) elements Ce through Lu, such information for the chloride, bromide, and iodide species is presently limited to the trihalides. In order to extend our understanding of the chemical bonding in these Ln halides, which involve filling of the 4f electron shells, it would be useful to have experimental values for the lower-valent Cl, Br, and I species. The thermochemical data will also be useful in the chemical modeling of metal halide lamp processes, where the more volatile Ln bromides and iodides are utilized. Kaledin et al.[1] recently have used a ligand field model to estimate the dissociation energies (D_0°) and ionization energies (IE) of the LnCl, LnBr, and LnI molecular species based on established values in the fluorides. In order to provide basic thermochemical data for halide lamp applications, we investigated gaseous equilibria in the Dy−Br and Dy−I systems using effusion-beam mass spectrometry, and results obtained for the monohalide, dihalide, and trihalide species are presented here.

Experimental Section

The gaseous species to be studied were generated by the reaction of $Br_2(g)$ or $I_2(g)$ with Dy(s) chips in a heated effusion cell reactor with a gas inlet system. A graphite effusion cell with a gas inlet tube was used for the Dy−Br system, while an identical molybdenum arrangement was used for the Dy−I system. In the Dy−Br studies, a few measurements on the Dy−Ag−Br system were made with Ag granules added to the cell containing Dy(s). The gaseous reagents were derived from Br_2(liq) and $I_2(s)$ samples vaporized from room temperature containers, and flow was controlled with a precision leak valve. Gaseous product species in the molecular effusion beam were ionized by electron impact, and the resulting ions were then drawn out, accelerated, mass analyzed in a magnetic field, and detected by a particle multiplier. The magnetic deflection instrument and molecular source arrangement have been described in earlier publications.[2−4] Cell heating was provided by a concentric tantalum spiral heater enclosed in radiation shields, and cell temperature was measured by optical pyrometry, sighting on a blackbody cavity in the cell lid. Threshold appearance potentials (APs) of observed ion signals were evaluated by the vanishing current method. Individual ion species and their neutral precursors were identified from the masses, isotopic patterns, and threshold APs of the observed ions. For use in evaluating reaction equilibrium constants, parent ion intensities were measured at 3 eV above threshold to minimize fragmentation effects. The pressure calibration factor required in one instance was obtained from measurements on the laboratory vapor pressure standards Ag, Au, and Sn; this calibration factor has been remarkably consistent over time. Measured ion signals were checked for their effusion cell origin by means of the characteristic neutral beam profiles. All aspects of the experimental technique and data interpretation are as described previously.[2−4] The Dy(s), Br_2(liq), and $I_2(s)$ samples were of reagent grade quality.

Results

A. Dy−Br System. At a cell temperature of 1360 K with $Br_2(g)$ addition, the beam signals Dy^+, $DyBr^+$, $DyBr_2^+$, $DyBr_3^+$ were observed with threshold APs of 5.8, 6.0, 7.0, and 10.1 eV, all ±0.3 eV, respectively. These values are in accord with the known and expected ionization energies (IE) of the corresponding parent neutrals, where the ionizing orbitals of the Dy−Br species change from essentially nonbonding Dy character in DyBr to increasing amounts of Br character as the second and third ligands are added. The observed threshold of $DyBr^+$ agrees with the IE(DyBr) estimate of 5.81 eV.[1] With elemental Ag addition to the cell, the additional ions Ag^+ and $AgBr^+$ were

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* Corresponding author. E-mail: donald.hildenbrand@sri.com.

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TABLE 1: Equilibrium Constants and Third-Law Enthalpy Changes for Gaseous Reactions 1−3[a]

T/K[b]	K_1[c]	$\Delta H^\circ_{298}(1)$ kcal mol^{-1}	K_2[c]	$\Delta H^\circ_{298}(2)$ kcal mol^{-1}	K_3[c]	$\Delta H^\circ_{298}(3)$ kcal mol^{-1}
1308 (2)					$(4.06 \pm 0.52) \times 10^{-4}$	17.5
1371 (3)	$(5.02 \pm 0.20) \times 10^{-2}$	15.0	$(2.97 \pm 0.30) \times 10^{-1}$	9.7		
1375 (2)					$(6.90 \pm 0.23) \times 10^{-4}$	17.0
1451 (3)	$(6.94 \pm 0.23) \times 10^{-2}$	15.0	$(3.23 \pm 0.26) \times 10^{-1}$	9.9		
1465					1.13×10^{-3}	16.6
1472 (3)	$(6.90 \pm 0.52) \times 10^{-2}$	15.2				
1492 (3)	$(1.06 \pm 0.08) \times 10^{-1}$	14.2	$(2.76 \pm 0.17) \times 10^{-1}$	10.6		
1534 (5)	$(7.11 \pm 0.92) \times 10^{-2}$	15.8	$(5.54 \pm 0.99) \times 10^{-1}$	8.7		
1541 (3)	$(1.09 \pm 0.07) \times 10^{-1}$	14.6	$(2.65 \pm 0.47) \times 10^{-1}$	11.0		
	av	15.0		10.0		17.0
	second law	15.6 ± 5		7.3 ± 5		25.1 ± 10

[a] Dy + $DyBr_2$ = 2DyBr (1); DyBr + $DyBr_3$ = $2DyBr_2$ (2); Ag + DyBr = AgBr + Dy (3). [b] Values in parentheses after listed T/Ks indicate the number of determinations at constant temperature. [c] Uncertainties in K values are average deviations from the mean of the several determinations.

observed, with threshold APs of 7.5 and 9.5 eV, respectively, indicating the presence of the species Ag and AgBr under those conditions. With the presence of neutral Dy, DyBr, $DyBr_2$, and $DyBr_3$ thus established, plus Ag and AgBr with Ag(s) addition, the equilibrium constants, K, of the gaseous reactions

$$\mathrm{Dy + DyBr_2 = 2DyBr} \qquad (1)$$

and

$$\mathrm{DyBr + DyBr_3 = 2DyBr_2} \qquad (2)$$

were evaluated from a series of parent ion intensity measurements made at ionizing energies of AP + 3 eV in the range of 1371−1541 K. A fragmentation correction, obtained from the mass spectrum of $DyBr_3$, was applied to $DyBr_3^+$ to give the total ion yield; none of the other signals required a correction. The ion intensity analogues of the K values for the isomolecular reactions 1 and 2 were used without further correction and are believed to be accurate within a factor of 2, based on past experience. A few measurements of the gaseous reaction equilibrium

$$\mathrm{Ag + DyBr = AgBr + Dy} \qquad (3)$$

were made over the range of 1308−1465 K and evaluated in similar fashion.

Third-law analysis yielded the reaction enthalpies $\Delta H^\circ_{298}(1) = 15.0$ kcal mol^{-1} and $\Delta H^\circ_{298}(2) = 10.0$ kcal mol^{-1}, both ±2 kcal mol^{-1}, and $\Delta H^\circ_{298}(3) = 17.0 \pm 2.5$ kcal mol^{-1}. The equilibrium data and derived results are summarized in Table 1. Note that most of the reported K values for reactions 1, 2, and 3 are the averages of several measurements at constant temperature; replicate measurements at a given temperature were made at constant Br_2 pressure. Thermal functions used in these and other equilibrium analyses were taken from sources described in the Appendix. Stated uncertainties are believed to reflect potential errors in both the equilibrium constants and thermal functions of the species involved. Although no attempt was made to do a careful study of the temperature dependence, Table 1 shows that, within their broader uncertainty limits, the second-law enthalpies are in relatively good agreement with third-law values. From the derived third-law enthalpies, one evaluates the Dy−Br bond dissociation energies (BDE) at 298 K as D(Dy−Br) = 83.7 ± 3.2 kcal mol^{-1}, D(BrDy−Br) = 98.7 ± 3.8 kcal mol^{-1}, and D(Br_2Dy−Br) = 108.6 ± 4.3 kcal mol^{-1}. The significance of these thermochemical results and the relation to information in the literature will be discussed below.

B. Dy−I System. With I_2(g) admitted to the cell at 1350 K, the effusion-beam signals Dy^+, DyI^+, DyI_2^+, DyI_3^+, and I^+ were observed with threshold APs of 6.0, 6.0, 7.7, 9.0, and 10.2 eV, indicating all to be parent ions in the initial threshold region. Again, the observed threshold of DyI^+ is in good accord with the IE(DyI) estimate of 5.88 eV by Kaledin et al.[1] As with the Dy−Br system, all of the corresponding parent neutral species are indicated to be present in the beam under these conditions. In similar fashion, parent ion intensities measured at AP + 3 eV were used to evaluate the K values for the gaseous reactions

$$\mathrm{Dy + DyI_2 = 2DyI} \qquad (4)$$

$$\mathrm{DyI + DyI_3 = 2DyI_2} \qquad (5)$$

$$\mathrm{Dy + I = DyI} \qquad (6)$$

in the range of 1311−1426 K. For reaction 6, a pressure calibration constant evaluated as described earlier, along with estimated ionization cross sections, was used in deriving absolute values of K from the intensity data. Again, a fragmentation correction was applied to the DyI_3^+ signal to obtain the DyI_3 total ion yield.

A summary of the derived K values and the calculated third-law reaction enthalpies is given in Table 2. Note that here, also, most of the listed K values are averages of several determinations at constant temperature. For two of the measurements at 1402 K the I_2 pressure was reduced by a factor of 2, with no change in the derived K values; all other measurements were made at constant I_2 pressure. Thermal functions used in the third-law calculations were evaluated as described in the Appendix. Third-law analysis gave the reaction enthalpies $\Delta H^\circ_{298}(4) = 15.8$ kcal mol^{-1}, $\Delta H^\circ_{298}(5) = 2.7$ kcal mol^{-1}, and $\Delta H^\circ_{298}(6) = -64.3$ kcal mol^{-1}, all ±2 kcal mol^{-1}. Again, second-law enthalpies are seen in Table 2 to be in relatively good agreement. The corresponding BDEs at 298 K derived from these results are D(Dy−I) = 64.3 ± 2 kcal mol^{-1}, D(IDy−I) = 80.1 ± 2.8 kcal mol^{-1}, and D(I_2Dy−I) = 82.8 ± 3.5 kcal mol^{-1}.

Discussion

Kaledin et al.[1] estimated the dissociation energies of the diatomic molecules LnCl, LnBr, and LnI using a ligand field model fitted to known values for most of the LnF species. Trends in the D°_0 values of the lanthanide monofluorides stretching from CeF to LuF are anchored by the established value for the preseries member BaF; it is assumed that similar trends will follow for the diatomic Ln chlorides, bromides, and

TABLE 2: Equilibrium Constants and Third-Law Enthalpy Changes for Gaseous Reactions 4−6[a]

T/K[b]	K_4[c]	$\Delta H^\circ_{298}(4)$ kcal mol^{-1}	K_5[c]	$\Delta H^\circ_{298}(5)$ kcal mol^{-1}	K_6,[c] atm^{-1}	$\Delta H^\circ_{298}(6)$ kcal mol^{-1}
1311	2.56×10^{-2}	15.9			2.66×10^{6}	−64.3
1314	2.55×10^{-2}	16.0			2.66×10^{6}	−64.4
1355 (2)	$(3.36 \pm 0.07) \times 10^{-2}$	15.7	(3.72 ± 0.04)	2.5	$(1.43 \pm 0.08) \times 10^{6}$	−64.8
1402 (5)	$(3.99 \pm 0.20) \times 10^{-2}$	15.8	(3.41 ± 0.20)	2.8	$(4.54 \pm 0.19) \times 10^{5}$	−63.8
1426 (2)	$(4.32 \pm 0.06) \times 10^{-2}$	15.8	(3.47 ± 0.05)	2.7	$(3.31 \pm 0.12) \times 10^{5}$	−64.1
	av	15.8		2.7		−64.3
	second law	17.4 ± 5		−2.3 ± 5		−69.3 ± 5

[a] Dy + DyI_2 = 2DyI (4); DyI + DyI_3 = $2DyI_2$ (5); Dy + I = DyI (6). [b] Values in parentheses after listed T/Ks indicate the number of determinations at constant temperature. [c] Uncertainties in K values are average deviations from the mean of the several determinations.

iodides which are likewise anchored by published values for BaCl, BaBr, and BaI.[1] The value D°_0(BaI) = 76.3 ± 1.4 kcal mol^{-1}selected by Kaledin et al.[1] appears reliable and leads to their estimated value D°_0(DyI) = 65.7 ± 1.4 kcal mol^{-1}, in reasonable accord with our experimental value of 63.8 ± 2 kcal mol^{-1}. However, their selected value D°_0(BaBr) = 85.5 ± 2.3 kcal mol^{-1} now appears several kcal mol^{-1} low because a more reliable determination[5] of D°_0 for the reference partner AlBr now yields the corrected value D°_0(BaBr) = 87.7 ± 2.2 kcal mol^{-1} from the mass spectrometric equilibrium measurement.[6] In addition, the more recent critical compilation of Gurvich et al.[7] selects D°_0(BaBr) = 88.4 ± 2.4 kcal mol^{-1} as the present best value, which then increases the D°_0(DyBr) estimate of Kaledin et al.[1] from 74.9 to 77.9 ± 2.3 kcal mol^{-1}, compared to our experimental value of D°_0(DyBr) = 83.1 ± 3.2 kcal mol^{-1}. These two values, 77.9 and 83.1 kcal mol^{-1}, are compatible within the combined uncertainties, and we are inclined to select the average of the two, D°_0(DyBr) = 80.5 ± 2.5 kcal mol^{-1} as present best value. This seems justified because of the relatively small number of equilibrium measurements on reaction 3 and the somewhat larger uncertainty in the dissociation energy of the reference partner AgBr.

With this new selected value of D°_0(DyBr), we revise our initial BDE values at 298 K for the Dy−Br system to D(Dy−Br) = 81.1 ± 2.5 kcal mol^{-1}, D(BrDy−Br) = 96.1 ± 3.2 kcal mol^{-1}, and D(Br_2Dy−Br) = 106.0 ± 3.8 kcal mol^{-1}. The selected BDE values at 298 K for the Dy−I system remain unchanged at D(Dy−I) = 64.3 ± 2 kcal mol^{-1}, D(IDy−I) = 80.1 ± 2.8 kcal mol^{-1}, and D(I_2Dy−I) = 82.8 ± 3.5 kcal mol^{-1}. Because the enthalpies of sublimation of $DyBr_3$(s) and DyI_3(s) may be evaluated from the vapor pressure data on these phases, and the enthalpies of formation of the solid phases are known, the atomization enthalpies of the two gaseous trihalides can be evaluated and compared with the sum of the three individual BDEs. The sources of these data are described in the Appendix. For $DyBr_3$(g), ΔH°_{298}(atomization) = 279.7 ± 2.1 kcal mol^{-1}, compared to the sum of the three selected Dy−Br BDEs at 283.2 ± 5.5 kcal mol^{-1}. And for DyI_3(g), ΔH°_{298}(atomization) = 224.2 ± 3 kcal mol^{-1}, while the sum of the individual BDEs is 227.2 ± 4.9 kcal mol^{-1}. For both $DyBr_3$ and DyI_3, the agreement between the results of these two independent routes to ΔH°_{298}(atomization) is quite satisfactory, indicating that the selected results are internally consistent. The selected atomization enthalpy of $DyBr_3$, 279.7 kcal mol^{-1}, is in accord with the upper bound of 329 kcal mol^{-1} derived by Hilpert et al.[16] from the AP of Dy^+ formed from electron impact ionization of $DyBr_3$.

The selected BDE values for the Dy−Br and Dy−I species derived from this analysis are summarized in Table 3, along with the corresponding standard enthalpies of formation, $\Delta_f H^\circ_{298}$, of the monohalide, dihalide, and trihalide species. Note that for both Dy−Br and Dy−I the first metal−halogen bonds formed to make the diatomic halides are considerably weaker than the second and third bonds. This probably results from the promotion energy required to raise the nonbonding (Xe) $4f^{10}6s^2$ ground state of the Dy atom to the unpaired (Xe) $4f^{10}5d6s$ bonding state before bond formation occurs.

TABLE 3: Selected Bond Strengths and Standard Enthalpies of Formation

bond	D_{298} kcal mol^{-1}	species	$\Delta_f H^\circ_{298}$ kcal mol^{-1}
Dy−Br	81.1 ± 2.5	DyBr	13.8 ±2.5
BrDy−Br	96.1 ± 3.2	$DyBr_2$	−55.6 ± 3.2
Br_2Dy−Br	106.0 ± 3.8	$DyBr_3$	−131.4 ±2.5[a] −134.9 ± 5[b]
Dy−I	64.3 ± 2	DyI	29.4 ± 2
IDy−I	80.1 ± 2.8	DyI_2	−25.2 ± 2.8
I_2Dy−I	82.8 ± 3.5	DyI_3	−79.4 ± 2.5 [a] −82.5 ± 4.6 [b]

[a] From sublimation data. [b] From BDE data. D°_0(DyBr) = 80.5 ± 2.5 kcal mol^{-1} = 3.49 ± 0.11 eV. D°_0(DyI) = 63.8 ± 2 kcal mol^{-1} = 2.77 ± 0.09 eV.

TABLE 4: Molecular Constants of Dy−Br and Dy−I Species Used in Calculating Thermal Functions

species[a]	I	σ	ω_i cm^{-1}
DyBr	61.1×10^{-39} [b]	1	270
$DyBr_2$	5.63×10^{-112} [c]	2	270, 55, 250
$DyBr_3$	5.12×10^{-111} [c]	6	269, 45, 259(2), 67(2)
DyI	96.8×10^{-39} [b]	1	190
DyI_2	2.82×10^{-111} [c]	2	190, 40, 170
DyI_3	3.47×10^{-110} [c]	6	189, 32, 182(2), 47 (2)

[a] DyBr, DyI: $g_0 = 18$, $g_1 = 16$, $\epsilon_1 = 828$ cm^{-1}. $DyBr_2$, DyI_2: $g_0 = 17$. $DyBr_3$, DyI_3: $g_0 = 16$, $g_1 = 14$, $\epsilon_1 = 3460$ cm^{-1}. [b] Units of g cm^2. [c] Units of g^3 cm^6.

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Appendix

The auxiliary thermochemical data used in analysis of the experimental results were taken from sources described here. A summary of the molecular constants used in calculating thermal functions for the Dy−Br and Dy−I gaseous species is given in Table 4, in terms of the moments of inertia, I, the rotational symmetry number, σ, the fundamental vibrational frequency, ω, the degeneracy of the electronic state, g, and the energy of the electronic state, ϵ.

$DyBr_3$, DyI_3. All molecular constants were taken from the values given by Myers and Graves.[8] Combination of the sublimation enthalpies and the enthalpies of formation of the solid phases described below led to the corresponding values $\Delta_f H^\circ_{298}$($DyBr_3$,g) = −131.4 ± 2.5 kcal mol^{-1} and $\Delta_f H^\circ_{298}$(DyI_3,g) = −79.4 ± 2.5 kcal mol^{-1}.

DyBr, $DyBr_2$, DyI, DyI_2. A symmetrical bent structure with a bond angle of 110° was assumed for the dihalides. Internuclear distances were assumed to be the same as those in the trihalides, and vibrational frequencies were estimated from those of the trihalides given by Myers and Graves.[8] Electronic energies and degeneracies were taken from those of the monovalent and divalent gaseous ions.[9]

Dy, Ag, AgBr, I. All thermochemical data for gaseous Dy, Ag, and I were taken from the IVTANTHERMO database.[10] For AgBr, thermal functions were calculated from the spectroscopic constants given by Huber and Herzberg.[11] From the torsion vapor pressures of AgBr(l),[12] the enthalpy of sublimation of AgBr monomer was derived on the assumption of equal mol fractions of monomer and trimer in the vapor. This is consistent with measured values in AgCl and AgI vapors. Combination of $\Delta H°$(sub, AgBr) with the thermochemical properties of AgBr(s,l) yielded the dissociation energy $D^{\circ}_{298}(\mathrm{AgBr}) = 66.7 \pm 2$ kcal mol^{-1}.

$DyBr_3$(s), DyI_3(s). For use in evaluating the enthalpies of sublimation, thermal functions were evaluated as described by Myers and Graves.[13] The standard enthalpy of formation of $DyBr_3$(s) was taken from the measurements of Hurtgen et al.,[14] whereas that of DyI_3(s) is from the evaluation of Wagman et al.[15] Third-law treatment of the sublimation pressures of Hilpert et al.[16] for $DyBr_3$(s), and of Hirayama et al.[17] for DyI_3(s), then yielded the enthalpies of formation of the gaseous trihalides given above.

AgBr(s,l). Thermal functions and standard enthalpies of formation were taken from the compilation of Pankratz.[18]

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