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Standard enthalpies of formation of yttrium gallides by high temperature reaction calorimetry

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Abstract

The standard enthalpies of formation ($\Delta_f H^0_{298.15}$) of Y_5Ga_3, YGa and YGa_2 at 298.15 were determined by high temperature liquid gallium solution calorimetry to be -59.6 ± 3.6, -68.9 ± 2.8 and -64.4 ± 2.5 kJ gatom^{-1} respectively. The enthalpy of formation of YGa_2 at 1125 K was determined by using precipitation calorimetry, to be -61.8 ± 2.8. The partial enthalpy of solution of yttrium in liquid gallium at infinite dilution ($\Delta\bar{H}^{\infty}_{Y}$) was determined to be -184.4 ± 5.3 kJ gatom^{-1}. © 2000 Elsevier Science S.A. All rights reserved.

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1. Introduction

The intermetallic compounds formed by rare earth elements are of particular interest regarding their potential usage as high value functional materials, such as permanent magnets and hydrogen storage material. In the pyrochemical processes used for reprocessing of spent nuclear fuels, it is necessary to recover the actinides and rare earths left in the spent molten salt after the completion of the process. For recovering the actinides and rare earths, the spent molten salt is equilibrated with calcium–gallium alloys [1]. Calcium acts as the reductant whereas gallium forms an alloy with the actinide and rare earth metals, to lower their activity, thus providing an additional driving force for the reduction reaction. Hence thermodynamic data on actinide–gallium and rare earth–gallium systems are of interest in pyrochemical processes. We have taken up systematic investigations on rare earth–gallium systems and reported our calorimetric measurements on Ce–Ga alloys in a subsequent paper [2]. In this paper, we report the results of our calorimetric studies on Y–Ga alloys.

Information on the binary phase diagram of Y–Ga system and the structures of the considered phases is available in the literature [3]. The phase diagram of the Y–Ga system shows three intermetallic compounds, the incongruently melting phase Y_5Ga_3, and the congruently melting phases YGa and YGa_2. Merker [4] has determined the enthalpies of formation of intermetallic compounds Y_5Ga_3, YGa and YGa_2 by solution calorimetry. Yamshchikov et al. [5] have determined the solubility of yttrium in gallium by using molten salt emf method and reported the Gibbs free energy of formation of YGa_2. Very recently, Meschel and Kleppa [6] have measured the standard enthalpies of formation of YGa and YGa_2 by direct synthesis calorimetry. In the present study, we have adopted two different calorimetric methods to determine the enthalpies of formation of the intermetallic compounds in the Y–Ga system. The standard enthalpies of formation of the intermetallic compounds Y_5Ga_3, YGa and YGa_2 at 298.15 K were determined by high-temperature gallium solution calorimetry. The enthalpy of formation of YGa_2 at 1125 K was derived from the measurements of the integral enthalpies of formation of Y–Ga alloys over the biphasic region $\{Y\text{–}Ga\}+\langle YGa_2\rangle$. In this paper, these calorimetric results are discussed.

2. Experimental

2.1. Synthesis of alloys

High purity yttrium (99.9%) from M/s. Aldrich, USA, and gallium (99.9%) from Nuclear Fuel Complex, India, were used for the preparation of the alloy samples. The intermetallic compounds, Y_5Ga_3, YGa and YGa_2 were

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prepared by arc melting stoichiometric amounts of Y with Ga in an arc melting setup. The arc-melted buttons were turned over and remelted several times in order to obtain a homogeneous phase. The buttons thus prepared were wrapped in tantalum foils, sealed in quartz tubes under argon atmosphere at sub ambient pressures and annealed. The Y_5Ga_3, YGa and YGa_2 buttons were annealed at 955 K, 982 K and 1073 K respectively for 360 h. The XRD patterns (Fig. 1) of the annealed samples confirmed the presence of the respective single-phase intermetallic compound. The rare earth metals and their alloys were stored and handled in an argon atmosphere glove box before calorimetric measurements.

2.2. Equipment

The calorimeter used in this work is a commercial high temperature heat-flow calorimeter of differential type supplied by SETARAM (Model HT1500) Lyon, France. The calorimeter has been described previously [7] and used extensively to determine the enthalpies of formation of uranium aluminides by Nagarajan et al. [8] and uranium and thorium gallides by Prabhakara Reddy et al. [9,10]. A PC based data acquisition system has been added recently to the calorimeter. The emf output of the thermopile detector of the calorimeter directly read on a digital multimeter (HP 34970A) is to a PC to generate the $\Delta T - t$ curves for the measurements.

2.3. Calibration

The calorimeter was calibrated at the end of each measurement series by dropping known amount of α-alumina (SRM 720 from NIST [11]) samples from the ambient temperature into the calorimeter at the experimental temperature, the corresponding enthalpy increments being taken from the work of NIST group [12].

2.4. Procedure

2.4.1. Solution calorimetry

In a typical solution calorimetric experiment, ~1.5 g of gallium was taken in an alumina crucible which was loaded into the calorimeter and heated to the temperature of measurement. Samples, each weighing ~10–20 mg and initially maintained at the ambient temperature, were dropped into the liquid gallium in the calorimeter. The amounts of solutes and the gallium solvent were so chosen as to form dilute solutions of solutes in gallium. The calorimetric signals recorded corresponded to the thermal effects of dissolution of the respective solutes in liquid gallium at the temperature of measurement. The thermal effects of dissolutions of Y, Y_5Ga_3, YGa and YGa_2 at the same experimental temperature were measured in separate experiments by using the above procedure.

2.4.2. Precipitation calorimetry

The experimental procedure is similar to the one used for solution calorimetric measurements, but in these measurements, only up to certain concentrations of yttrium, the two metals readily mixed to form a single phase liquid alloy. Thereafter a solid intermetallic compound YGa_2 precipitated from the liquid alloy and the measurements were continued over the two phase region containing the liquid alloy {Y–Ga} and the solid intermetallic compound, YGa_2. The thermal effect corresponding to each calorimet-

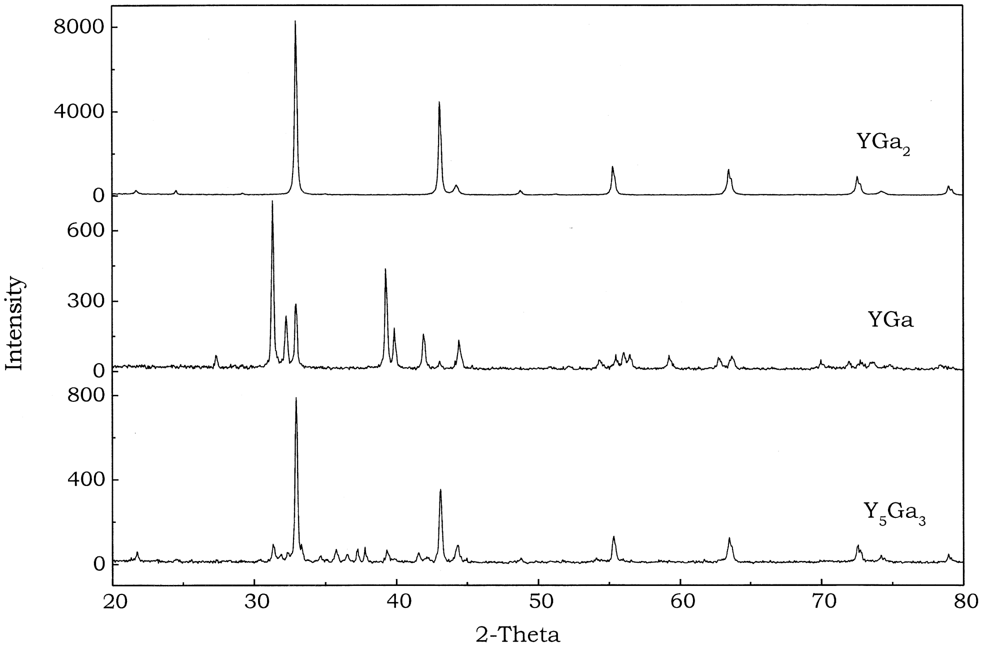

Fig. 1. X-ray diffraction patterns of Y–Ga alloys.

ric signal recorded in these measurements is the net result of two heat effects: (a) The endothermic effect due to the heating of yttrium from ambient to the temperature of measurement, 1125 K and its subsequent melting. (b) The exothermic effect due to the formation of Y–Ga alloy either from the elements or from an existing alloy of a different composition. From the measured thermal effects and the literature data [13] for the enthalpy of yttrium, the integral enthalpies of formation of the alloys in the two phase region were computed as a function of the overall composition of the alloy by using the method employed by Nagarajan et al. [8]. After the completion of the measurements, the final states of the contents of the crucible were examined by XRD. A small flow of high purity argon was maintained in the experimental chamber during the precipitation calorimetric measurements as in the case of solution calorimetric measurements.

3. Results

3.1. Enthalpy of formation at 298.15 K

The thermal effects of dissolution (Q^E) of Y in liquid gallium measured at 1099 K are given in Table 1. The thermal effects of dissolution of Y_5Ga_3 at 1103 K, YGa at 1095 K and those of YGa_2 at 1099 K in liquid gallium measured in separate experiments are given in Tables 2–4. The thermal effects of dissolution (Q^E) of Y, Y_5Ga_3, YGa and YGa_2 are all found to be independent of the concentration of the respective solutes in liquid gallium within experimental errors, as shown in these tables. Hence the

Table 1
Experimental data for the thermal effects of dissolution of yttrium in liquid gallium at 1099 K[a]

No.	Wt. of α-Al_2O_3 (mg)	Peak area (Counts)
Calibration:		
1	76.74	321
2	90.55	349
3	85.83	345
4	67.41	266
5	60.01	240

$\langle Y\rangle_{298.15}\rightarrow[Y]_{\{Ga,1099\}}$

No.	Wt. of Y (mg)	Peak area (Counts)	x_Y (at.%)	Q^E_Y (kJ gatom^{-1})
Measurement:				
1	11.71	−86	0.59	−144.82
2	10.80	−82	1.13	−149.72
3	11.02	−80	1.68	−143.15
4	12.26	−88	2.28	−141.54
5	11.98	−87	2.86	−143.20

$Q^{E,\infty}_Y = -144.49$ kJ gatom^{-1}

[a] Amount of gallium in the calorimeter = 1.54098 g ($H^0_T - H^0_{298.15}$) of α-Al_2O_3 at 1099 K = 90.4049 kJ mol^{-1}. Calibration constant = 0.22180±0.0066 J Count^{-1}. Overall uncertainty in $Q^{E,\infty}_Y = \pm 5.4$ kJ gatom^{-1}.

Table 2
Experimental data for the thermal effects of dissolution of Y_5Ga_3 in liquid gallium at 1103 K[a]

No.	Wt. of α-Al_2O_3 (mg)	Peak area (Counts)
Calibration:		
1	94.18	2183
2	90.17	2100
3	47.71	1038
4	77.68	1744
5	53.36	1163

$\langle Y_{5/8}Ga_{3/8}\rangle_{298.15}\rightarrow[5/8\ Y+3/8\ Ga]_{\{Ga,1103\}}$

No.	Wt. of sample (mg)	Peak area (Counts)	x_Y (at.%)	$Q^E_{Y_{5/8}Ga_{3/8}}$ (kJ gatom^{-1})
Measurement:				
1	12.64	−78	0.43	−20.00
2	14.56	−101	0.92	−22.49
3	13.90	−92	1.39	−21.45
4	15.48	−96	1.89	−20.10
5	11.42	−67	2.26	−19.01

$Q^{E,\infty}_{Y_{5/8}Ga_{3/8}} = -20.61$ kJ gatom^{-1}

[a] Amount of gallium in the calorimeter = 1.54535 g ($H^0_T - H^0_{298.15}$) of α-Al_2O_3 at 1103 K = 90.9120 kJ mol^{-1}. Calibration constant = 0.03967±0.0013 J Count^{-1}. Overall uncertainty in $Q^{E,\infty}_{Y_{5/8}Ga_{3/8}} = \pm 1.5$ kJ gatom^{-1}.

Table 3
Experimental data for the thermal effects of dissolution of YGa in liquid gallium at 1095 K[a]

No.	Wt. of α-Al_2O_3 (mg)	Peak Area (Counts)
Calibration:		
1	48.13	1023
2	54.70	1223
3	55.79	1192
4	57.33	1271
5	77.84	1745

$\langle Y_{1/2}Ga_{1/2}\rangle_{298.15}\rightarrow[1/2\ Y+1/2\ Ga]_{\{Ga,1095\}}$

No.	Wt. of sample (mg)	Peak Area (Counts)	x_Y (at.%)	$Q^E_{Y_{1/2}Ga_{1/2}}$ (kJ gatom^{-1})
Measurement:				
1	24.85	76	0.73	9.77
2	17.31	57	1.22	10.51
3	20.72	73	1.80	11.25
4	19.49	52	2.33	8.52
5	19.56	66	2.86	10.77

$Q^{E,\infty}_{Y_{1/2}Ga_{1/2}} = 10.16$ kJ gatom^{-1}

[a] Amount of gallium in the calorimeter = 1.47885 g ($H^0_T - H^0_{298.15}$) of α-Al_2O_3 at 1095 K = 89.9003 kJ mol^{-1}. Calibration constant = 0.04026±0.0010 J Count^{-1}. Overall uncertainty in $Q^{E,\infty}_{Y_{1/2}Ga_{1/2}} = \pm 1.0$ kJ gatom^{-1}.

Table 4
Experimental data for the thermal effects of dissolution of YGa_2 in liquid gallium at 1099 K[a]

No.	Wt. of α-Al_2O_3 (mg)	Peak area (Counts)
Calibration:		
1	93.57	396
2	93.47	390
3	75.29	337
4	164.45	690
5	102.4	450

$\langle Y_{1/3}Ga_{2/3}\rangle_{298.15} \rightarrow [1/3\ Y + 2/3\ Ga]_{\{Ga,1099\}}$

No.	Wt. of sample (mg)	Peak Area (Counts)	x_Y (at.%)	$Q^{E}_{Y_{1/3}Ga_{2/3}}$ (kJ gatom^{-1})
Measurement:				
1	13.52	29	0.27	33.74
2	7.79	18	0.42	36.34
3	7.07	15	0.56	33.37
4	7.67	16	0.70	32.81
5	7.60	17	0.85	35.18

$Q^{E,\infty}_{Y_{1/3}Ga_{2/3}} = 34.29$ kJ gatom^{-1}

[a] Amount of gallium in the calorimeter = 1.53450 g $(H^0_T - H^0_{298.15})$ of α-Al_2O_3 at 1099 K = 90.4049 kJ mol^{-1}. Calibration constant = 0.20664 ± 0.0064 J Count^{-1}. Overall uncertainty in $Q^{E,\infty}_{Y_{1/3}Ga_{2/3}} = \pm$ 1.8 kJ gatom^{-1}.

mean of the thermal effects of dissolution (Q^E) values were taken as ($Q^{E,\infty}$), the thermal effects of dissolution at infinite dilution. The overall uncertainties in the $Q^{E,\infty}$ values were computed by taking into account the standard deviation of the Q^E values and that of the calibration measurements and by invoking the error propagation theory. The thermal effects of dissolution at infinite dilution, ($Q^{E,\infty}$) along with the standard deviation are given in the respective tables. From the thermal effects of dissolution at infinite dilution of Y, Y_5Ga_3, YGa and YGa_2, the standard enthalpies of formation of Y_5Ga_3, YGa and YGa_2 were computed by using the following equation:

$$\Delta_f H^0_{298.15}\langle Y_x Ga_{(1-x)}\rangle = xQ^{E,\infty}_{Y} + (1-x)Q^{E,\infty}_{Ga} - Q^{E,\infty}_{Y_x Ga(1-x)} \tag{1}$$

In the above equation, the term $Q^{E,\infty}_{Ga}$ corresponds to the enthalpy increment of $(H^0_T - H^0_{298.15})$ Ga, which was taken from the literature [13]. For the determination of enthalpy of formation of the intermetallic compounds by solution calorimetry, it is necessary to measure the enthalpies of dissolution of the pure components and of the intermetallic compounds in the same metallic bath and at the same temperature. In our solution calorimetric measurements, the dissolution experiments were performed at 1103 K and 1095 K for the alloys, whereas the enthalpy of dissolution of yttrium metal were performed at 1099 K. Though the temperatures of the dissolution experiments were different for the alloys and the component metal yttrium, no correction was applied for the difference in temperatures, since it was reasonably presumed that the partial enthalpies at infinite dilution of Y in Ga do not vary within the considered temperature range. The enthalpies of formation of Y_5Ga_3, YGa and YGa_2 at 298.15 thus obtained are given in Table 7 along with the literature data.

3.2. Enthalpy of formation at 1125 K by precipitation calorimetry

The integral enthalpies of formation of Y–Ga alloys measured at 1125 K by precipitation calorimetric measurements are given in Tables 5 and 6. These values are plotted as a function composition in Fig. 2. At 1125 K, the measured integral enthalpies up to 9.3 at.% Y covers the single-phase liquid region and then the two-phase region containing $\{Y\text{–}Ga\} + \langle YGa_2\rangle$. The integral enthalpy of formation of the alloy with the composition $Y_{1/3}Ga_{2/3}$, obtained from the integral enthalpies of formation of alloys in the two-phase region, corresponds to the enthalpy of the following reaction at 1125 K which is −65.54 kJ gatom^{-1}.

$$1/3\langle Y\rangle + 2/3\{Ga\} \rightarrow \langle Y_{1/3}Ga_{2/3}\rangle \tag{2}$$

From the enthalpy of the above reaction, the enthalpy of

Table 5
Integral enthalpies of formation of Y–Ga alloys at 1125 K (RUN I)[a]

No.	Wt. of α-Al_2O_3 (mg)	Peak area (Counts)
Calibration:		
1	47.96	1053
2	77.58	1703
3	53.33	1136
4	93.35	2105
5	57.26	1190

No.	Wt. (mg)	Peak area (Counts)	x_Y (at.%)	$-\Delta H$ (kJ gatom^{-1})
Measurement:				
1	49.67	−3037	2.62	7.06
2	41.21	−4265	4.69	16.02
3	41.10	−4639	6.67	25.32
4	40.22	−3384	8.52	31.90
5	20.25	−1057	9.43	33.93
6	15.80	−940	10.13	35.70
7	49.15	−1228	12.23	38.00
8	51.54	−1432	14.32	40.55
9	33.54	−1319	15.64	42.81
10	42.20	−1022	17.23	44.49
11	53.21	−1480	19.16	46.83
12	43.81	−1418	20.68	48.99
13	44.23	−1041	22.16	50.48

[a] Measurements up to 9.3 at.% Y (5th sample) are in {Y–Ga} and above 9.3 at.% Y are over $\{Y\text{–}Ga\} + \langle YGa_2\rangle$ two-phase region. Amount of gallium in the calorimeter = 1.44895 g $(H^0_T - H^0_{298.15\ K})$ of α-Al_2O_3 at 1125 K = 93.1060 kJ mol^{-1}. Calibration constant = 0.04215 ± 0.0013 J Count^{-1}.

Table 6
Integral enthalpies of formation of Y–Ga alloys at 1125 K (RUN II)[a]

No.	Wt. of α-Al_2O_3 (mg)	Peak area (Counts)
Calibration:		
1	77.75	1652
2	48.03	1703
3	94.02	2127
4	57.38	1177
5	53.29	1130

No.	Wt. (mg)	Peak area (Counts)	x_Y (at.%)	$-\Delta H$ (kJ $gatom^{-1}$)
Measurement:				
1	40.89	−2537	2.11	5.78
2	41.32	−4235	4.15	14.55
3	40.95	−4599	6.09	23.64
4	31.23	−2408	7.52	28.31
5	30.25	−1557	8.86	31.30
6	25.80	−1450	9.97	34.00
7	48.06	−1264	11.98	36.36
8	40.15	−1423	13.59	38.87
9	32.35	−1302	14.84	41.09
10	42.12	−1033	16.42	42.80
11	43.29	−1485	17.98	45.17
12	45.81	−1400	19.57	47.31
13	40.53	−1041	20.93	48.83
14	46.73	−1638	22.44	51.20

[a] Measurements up to 9.3 at.% Y (6th sample) are in {Y–Ga} and above 9.3 at.% Y are over {Y–Ga}+⟨YGa_2⟩ two-phase region. Amount of gallium in the calorimeter=1.48951 g ($H^0_T - H^0_{298.15\text{ K}}$) of α-$Al_2O_3$ at 1125 K=93.1060 kJ mol^{-1}. Calibration constant=0.04233±0.0017 J $Count^{-1}$.

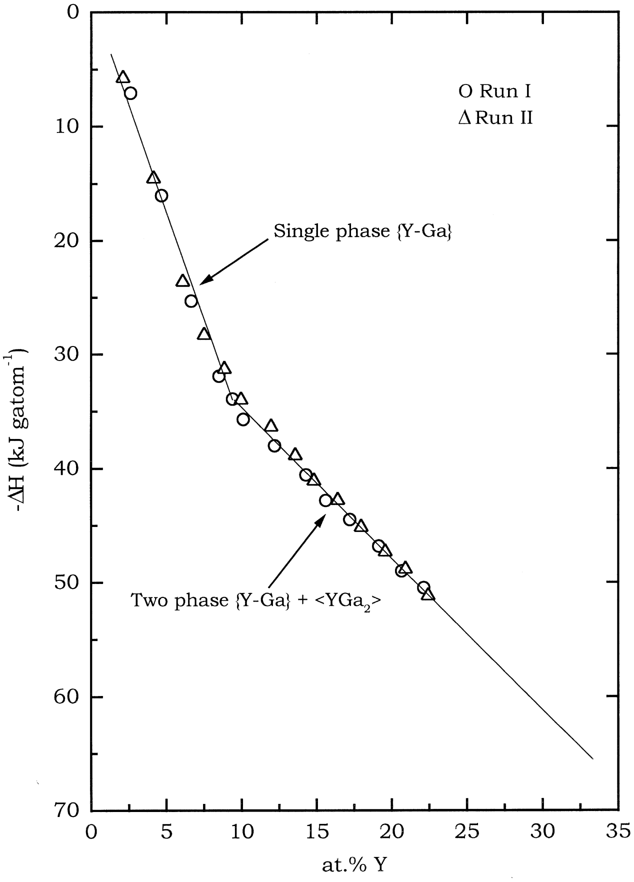

Fig. 2. Integral enthalpies of formation of Y–Ga alloys at 1125 K.

formation of the intermetallic compound $Y_{1/3}Ga_{2/3}$ with respect to solid gallium, corresponding to the reaction,

$$1/3\langle Y\rangle + 2/3\langle Ga\rangle \rightarrow Y_{1/3}Ga_{2/3}\rangle \quad (3)$$

was then computed by using the literature data [14] for the enthalpy of fusion of gallium. The standard enthalpy of formation thus obtained was −61.81±2.8 kJ $gatom^{-1}$.

3.3. Partial enthalpy of solution of yttrium

The partial enthalpy of dissolution of Y in gallium at infinite dilution ($\Delta\bar{H}_Y^\infty$) was computed from the measured thermal effects of dissolution at infinite dilution ($Q_Y^{E,\infty}$) of Y by using the following equation:

$$\Delta\bar{H}_Y^\infty = Q_Y^{E,\infty} - [(H^0_{1099} - H^0_{298.15}) + \Delta_{\text{trans}}H^0 + \Delta_{\text{fus}}H^0]_Y \quad (4)$$

Since the experimental temperature is below the melting temperature (1799 K), as well as the transition temperature (1752 K) of Y, the corresponding enthalpy values were incorporated in the above equation to compute the partial enthalpy of solution. The data for the enthalpy increment, the enthalpy of fusion and the enthalpy of transformation of Y were taken from the literature [13]. The computed partial enthalpy of solution of Y is given in Table 8.

4. Discussion

In Table 7, we compare our values of enthalpies of formation obtained by solution as well as precipitation calorimetry with the literature values. The present enthalpy of formation of YGa_2 at 1125 K obtained by us from precipitation calorimetry, is in very good agreement, within error limits, with our value at 298.15 K, obtained by solution calorimetry, but these values are slightly less exothermic than the calorimetric values of Merker et al. [4]. Our solution calorimetric value is in agreement with that of Meschel and Kleppa [6] within the uncertainty limits. The present values for the enthalpy of formation of YGa_2 are slightly less exothermic than the enthalpies of formation of Yamshchikov et al. [5] from molten salt emf measurements. The enthalpy data for YGa_2 from our calorimetric measurements are considered more reliable than the literature data because they have been obtained by two different methods, which are in very good agreement.

The present enthalpy of formation of Y_5Ga_3 at 298.15 K obtained by solution calorimetry is in very good agreement

Table 7
Standard enthalpies of formation of Y–Ga alloys

Compound	$-\Delta_f H^0_{298.15}$ (kJ gatom^{-1})	Method	Reference
Y_5Ga_3	59.6±3.6	Ga. Soln. Calori.: 1103 K	This work
	57.0	Ga. Soln. Calori.: 1176 K	[4]
	61.0	Miedema's Model	[14]
YGa	68.9±2.8	Ga. Soln. Calori.: 1095 K	This work
	71.0	Ga. Soln. Calori.: 1176 K	[4]
	71.4±1.7	Dir. Syn. Calori.: 1373 K	[6]
	73.0	Miedema's Model	[14]
YGa_2	64.4±2.5	Ga. Soln. Calori.: 1099 K	This work
	61.8±2.8	Precipitation Calori.: 1125 K	This work
	69.0	Ga. Soln. Calori.: 1176 K	[4]
	68.1±2.3	Dir. Syn. Calori.: 1373 K	[6]
	70.9±1.5	EMF: 650–854 K	[5]
	68.4±1.3	EMF: 854–1150 K	[5]
	65.0	Miedema's Model	[14]

Table 8
Partial enthalpy of solution of yttrium in liquid gallium at infinite dilution

$-\Delta\bar{H}\infty$ (kJ gatom^{-1})	Method	Temperature (K)	Reference
184.4±5.3	Soln. Calori.	1099	This work
174.4	Soln. Calori.	1176	[4]
175.0±1.8	EMF	650–854	[5]
167.0±1.8	EMF	854–1150	[5]
180.25	Miedema's model	14	

with the data of Merker et al. [4] also obtained by solution calorimetry. The present enthalpy of formation of YGa is also in agreement with those of Merkar et al. [4] obtained by solution calorimetry as well as that of Meschel and Kleppa [6] by direct synthesis calorimetry. The present values for the enthalpies of formation of Y_5Ga_3, YGa and YGa_2 are in very good agreement with the values computed using Miedema's model [14]. The enthalpies of formation of yttrium gallides are shown as a function of yttrium concentration in the Y–Ga system in Fig. 3. The results of the present study show similar trends as shown by the literature data [4,6,14].

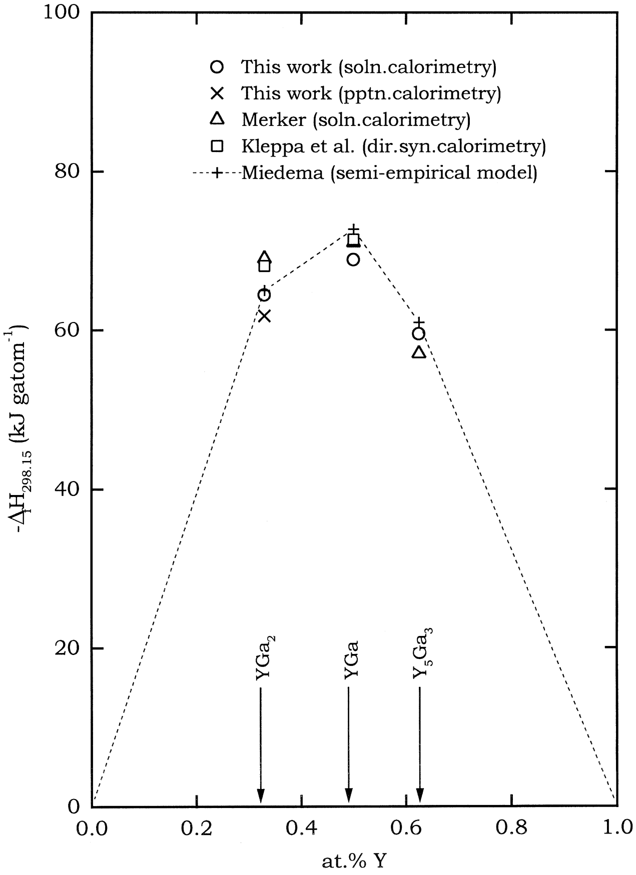

Fig. 3. Enthalpy of formation as a function of composition in the Y–Ga system.

In Table 8, the partial enthalpy values of Y in gallium at infinite dilution are compared with the literature values. Our partial enthalpy value for Y is slightly more exothermic than those of calorimetric as well as emf measurements of previous investigators, but it is in good agreement with the Miedema's value [14].

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