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The standard molar enthalpies of formation of $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ by acid solution calorimetry

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ABSTRACT

The standard molar enthalpies of formation, $\Delta_f H_m^0$ (298.15 K) of $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ have been determined using acid solution calorimetry. The enthalpies of solution of the compounds $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$, as well as those of mixtures of $\text{Fe}_2\text{O}_3(\text{s})$ and $\text{Pb}_3\text{O}_4(\text{s})$ in $\text{HCl}(\text{aq}, 6 \text{ mol}\cdot\text{dm}^{-3})$ at 298.15 K were measured. Using these values, the standard enthalpies of formation ($\Delta_f H_m^0$) of $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ were determined as $-(1324.2 \pm 11.1) \text{ kJ}\cdot\text{mol}^{-1}$ and $-(2347.8 \pm 10.7) \text{ kJ}\cdot\text{mol}^{-1}$, respectively.

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1. Introduction

Lead and lead-bismuth eutectic alloy are considered as suitable candidate coolants for the Accelerator Driven Sub-critical Systems (ADS) due to their desirable physical and chemical properties [1]. However, these coolants are highly corrosive towards the structural materials and this poses a critical challenge for their use. One of the approaches to reduce the corrosion is controlling the oxygen concentration thereby forming a protective oxide film on the surface of the structural steels. Such technique was first developed and deployed in Russia [2–4] and is being studied extensively worldwide [5,6]. The oxide film effectively separates the base material from LBE and the direct dissolution of the steel becomes negligible as the diffusion coefficients of the alloying components of steel in the oxide are very low [7,8]. Thermochemical data on Pb-M-O and Bi-M-O (M = alloying components in steel) systems are needed to understand the formation and stability of the oxide film over the steel surface in the lead and LBE coolant circuit. Phase diagram of Pb-Fe-O system has been established recently and this system has three ternary compounds namely $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$, $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ and $\text{PbFe}_{12}\text{O}_{19}(\text{s})$ [9]. This paper presents the enthalpy of formation of $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ determined using acid solution calorimetry.

2. Experimental

2.1. Chemicals

$\text{Fe}_2\text{O}_3(\text{s})$ powder (mass fraction purity > 0.9999 on metal basis, M/s Alfa Aesar, UK), $\text{Pb}_3\text{O}_4(\text{s})$ (mass fraction purity > 0.99 on metal basis, M/s Aldrich Chem. Co, USA) and $\text{PbO}(\text{s})$ powder (mass fraction purity > 0.999 on metal basis, M/s Aldrich Chem. Co., USA) were the starting reagents used. Thermogravimetry experiments coupled with evolved gas analysis showed that $\text{PbO}(\text{s})$ and $\text{Fe}_2\text{O}_3(\text{s})$ contained ~1.37 wt% and ~0.17 wt% of moisture, respectively. It was also found that $\text{PbO}(\text{s})$ contained ~2 wt% lead carbonate impurity. The $\text{PbO}(\text{s})$ powder was therefore heated in argon atmosphere at 773 K for 2 h while the $\text{Fe}_2\text{O}_3(\text{s})$ powder was calcined in air at 523 K for 2 h to remove the moisture before using them in experiments. $\text{Fe}(\text{NO}_3)_3\cdot 9\text{H}_2\text{O}(\text{s})$ (mass fraction purity > 0.9999 on metal basis, M/s Aldrich Chem. Co., USA) was also used for preparing finely divided $\text{Fe}_2\text{O}_3(\text{s})$.

2.2. Compound preparation

The compounds, $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ were prepared by solid state reaction and solution route, respectively. $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ was prepared by the solid-state reaction of mixtures of appropriate molar ratios of lead and iron oxide powders. The mixtures were taken in the form of compacted pellets, heated in alumina crucibles in ambient air at 998 K for 50 h with one intermediate grinding and finally quenched in ice cold water. The compound ' $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ ' is non stoichiometric with its stoichiometry ranging from $\text{PbO}\cdot 2\text{Fe}_2\text{O}_3$ to $\text{PbO}\cdot 2.5\text{Fe}_2\text{O}_3$. However for the present work, the

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compound with exact composition of $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ was prepared by co-precipitation method and used. For this preparation, stoichiometric amounts of PbO and high purity iron powder (mass fraction purity > 0.999 on metal basis, M/s Alfa Aesar, UK) were first dissolved in minimum amount of nitric acid and the resultant nitrate solution was added to an excess amount of ammonium hydroxide solution. The precipitate formed was then filtered, dried, pelletized and heated in air at 1048 K for 24 h. The products obtained by all these methods of preparation were characterized by X-ray diffraction using Siemens D500 X-ray powder diffractometer with $\text{Cu K}\alpha$ radiation and graphite monochromator. The XRD patterns obtained for $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ matched with the patterns reported in JCPDS files for these compounds, viz., 33-0756 and 49-0753, respectively.

Commercially available $\text{Fe}_2\text{O}_3(\text{s})$ powders are usually sintered at high temperature and hence require prolonged time for their dissolution in the calorimetric solvent, namely HCl (aq, $6 \text{ mol}\cdot\text{dm}^{-3}$). To enhance the dissolution rate, fine $\text{Fe}_2\text{O}_3(\text{s})$ powder was prepared. This was achieved by dissolving high purity ferric nitrate in distilled water followed by precipitation as $\text{Fe}(\text{OH})_3$ by the addition of ammonium hydroxide solution. The precipitate was filtered, dried and heated at 623 K for 4 h in air and the product was analysed by XRD.

2.3. Calorimetric technique

The enthalpy of formation of $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ were determined employing acid solution calorimetry and the details of the experimental set up are described in our earlier publication [10]. The temperature of the water bath was maintained within $\pm 0.002 \text{ K}$ and the accuracy of measurement of temperature using a thermistor inside the calorimeter was within $\pm 0.001 \text{ K}$.

The performance of the calorimeter was tested with KCl (SRM 1655, M/s.N.B.S., USA) dissolution in 0.300 dm^3 double distilled water. The molar enthalpy of solution of KCl in distilled water at infinite dilution derived in this study was: $\Delta_{\text{sol}}H_m^\infty$ (298.15 K) for $\text{KCl} = 17.15 \pm 0.06 \text{ kJ}\cdot\text{mol}^{-1}$. This value agreed well with the value of $17.21 \pm 0.01 \text{ kJ}\cdot\text{mol}^{-1}$ reported by Venugopal et al. [11] and N.B.S. value of $17.241 \pm 0.018 \text{ kJ}\cdot\text{mol}^{-1}$ [12]. The calorimeter performance was also tested with tris(hydroxyl methyl) amino-methane (TRIS). The enthalpy of solution was measured by dissolving known amount of TRIS (0.997 mass fraction purity) from MERK, UK, in 100 cm^3 of $0.1 \text{ mol}\cdot\text{dm}^{-3}$ HCl (aq) and the results are given in table 1. The value of the molar enthalpy of solution of TRIS measured in the present work is $-29.99 \pm 0.30 \text{ kJ}\cdot\text{mol}^{-1}$ which is in agreement with the NIST value of $-29.770 \pm 0.032 \text{ kJ}\cdot\text{mol}^{-1}$ [13]. The results obtained for KCl and TRIS dissolution which is exothermic and endothermic, respectively, established the suitability of the experimental set up for calorimetric measurements. Measurements were also carried out to quantify the thermal energy released during the breaking of the bulb by a blank run. However, the heat energy released during the process was found to be within

TABLE 1
The enthalpy of solution $\Delta_{\text{sol}}H_m^\circ$ of TRIS(s) in 0.100 dm^3 of 0.1 M HCl at $T = 298.15 \text{ K}$ (Molar mass of TRIS: 121.1).

Run	Mass of TRIS / g (g)	Experimental enthalpy change, ΔH (J)	Molar enthalpy of solution, $\Delta_{\text{sol}}H_m^\circ$ ($\text{kJ}\cdot\text{mol}^{-1}$)
1	0.2046	-50.45	-29.86
2	0.2980	-74.18	-30.14
3	0.4013	-97.84	-29.53
4	0.4182	-104.62	-30.30
5	0.4014	-99.79	-30.11
Average:			$-(29.99 \pm 0.30)^a$

^a Uncertainty is the standard deviation from the mean.

the detection limit of temperature of the calorimeter ($\pm 0.001 \text{ K}$). It is to be pointed out that, the glass bulb was designed with ultra thin walled bulb which breaks completely into pieces with a little touch of the push rod, allowing the solutes to mix with the calorimetric solvent. In such case, the thermal energy associated with the breakage of the bulb containing the sample is expected to be negligible, which is evident from the calibration runs of KCl and TRIS in the respective solvents.

Calorimetric measurements involving samples of the lead ferrites, $\text{Fe}_2\text{O}_3(\text{s})$ and $\text{Pb}_3\text{O}_4(\text{s})$ were carried out in HCl (aq, $6 \text{ mol}\cdot\text{dm}^{-3}$). The sample for the experiment was weighed accurately, placed inside the sample tube along with a glass push rod and isolated the sample from ambient using an O-ring. These operations were carried out inside an argon atmosphere glove box. 0.300 dm^3 of HCl (aq, $6 \text{ mol}\cdot\text{dm}^{-3}$) was transferred into calorimetric vessel and equilibrated at the bath temperature. The calorimeter was fitted with its flange and all the components were allowed to equilibrate at the bath temperature with the stirrer rotating at a constant speed. When the temperature of the calorimeter was in equilibrium with the surrounding water bath, a steady temperature signal was obtained. Under these conditions, the bulb containing the sample was broken and allowed to dissolve in the solvent, and the calorimeter signal was recorded. The maximum time taken for dissolution of samples during these experiments was 7 min. The system was calibrated electrically in situ before and after each of this dissolution experiment. The temperature change ΔT during electrical calibration as well as during sample dissolution was corrected for heat exchange between calorimeter and surroundings by the method suggested by Kubaschewski and Alcock [14] and was used for the evaluation of the enthalpy change of the reaction. Experiments were repeated for obtaining the reproducibility.

TABLE 2

The standard molar enthalpies of solution of $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$, $\text{PbFe}_5\text{O}_{8.5}(\text{s})$, $\text{Fe}_2\text{O}_3(\text{s})$ and $\text{Pb}_3\text{O}_4(\text{s})$ in a volume 0.300 dm^3 of HCl (aq, $6 \text{ mol}\cdot\text{dm}^{-3}$) at $T = 298.15 \text{ K}$; m denotes the mass of sample, ΔH the experimental enthalpy change, $\Delta_{\text{sol}}H_m$ the molar enthalpy of solution, and M denotes the molar mass.

Sample	m / g	ΔH / J	$\Delta_{\text{sol}}H_m / (\text{kJ}\cdot\text{mol}^{-1})$
$\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ ($M: 606.069 \text{ g}\cdot\text{mol}^{-1}$)	0.1989	-88.4	-269.3
	0.3009	-138.1	-278.2
	0.3412	-153.4	-272.4
	0.1914	-83.1	-263.0
	Average $\Delta_{\text{sol}}H_m = -(270.7 \pm 6.3)^a$		
$\text{PbFe}_5\text{O}_{8.5}(\text{s})$ ($M: 622.465 \text{ g}\cdot\text{mol}^{-1}$)	0.0832	-24.1	-180.2
	0.0747	-21.9	-182.6
	0.1197	-34.5	-179.5
	0.0730	-21.3	-181.6
	Average $\Delta_{\text{sol}}H_m = -(181.0 \pm 1.4)^a$		
$\text{Pb}_3\text{O}_4(\text{s})$ ($M: 685.566 \text{ g}\cdot\text{mol}^{-1}$)	0.1966	-84.9	-296.2
	0.1836	-78.0	-291.2
	0.1372	-59.3	-296.1
	0.1583	-65.4	-283.4
	0.1862	-82.3	-303.1
Average $\Delta_{\text{sol}}H_m = -(294.0 \pm 7.3)^{a,b}$			
$\text{Fe}_2\text{O}_3(\text{s})$ ($M: 159.691 \text{ g}\cdot\text{mol}^{-1}$)	0.0595	-20.9	-56.1
	0.0623	-21.4	-54.8
	0.0659	-20.8	-50.3
	0.0662	-21.0	-50.7
	0.0878	-31.0	-56.4
	0.0811	-25.6	-50.4
Average $\Delta_{\text{sol}}H_m = -(53.1 \pm 3.0)^a$			

^a Uncertainty is the standard deviation from the mean.

^b Correction for the evolution of O_2 was calculated as follows: it was assumed that $\text{O}_2(\text{g})$ was saturated with HCl vapour at the partial pressures given in the International Critical Table [15]. The correction amounts to $0.032 \text{ kJ}\cdot\text{mol}^{-1}$, assuming that the stoichiometric amount of O_2 was evolved and was saturated with the solvent. We have no proof for this. Therefore the correction is given as uncertainty of $\pm 0.016 \text{ kJ}\cdot\text{mol}^{-1}$. However, these corrections are well within the uncertainty limit.

TABLE 3

Reaction scheme for the standard molar enthalpy of formation of $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$, $\Delta_f H_m^\circ$ at 298.15 K; ΔH denotes enthalpy of reaction $\Delta_f H_m^\circ$ ($\text{Pb}_2\text{Fe}_2\text{O}_5, \text{cr}$) = $\Delta H(4)$ = $\Delta H(1)$ + $\Delta H(2)$ + $\Delta H(3)$.

S. no.	Reaction		$\Delta H_m/(\text{kJ}\cdot\text{mol}^{-1})$
1	$(2/3)\text{Pb}_3\text{O}_4(\text{s}) + \text{Fe}_2\text{O}_3(\text{s}) = \text{Pb}_2\text{Fe}_2\text{O}_5(\text{s}) + (1/3)\text{O}_2$	$\Delta H(1)$	$-(21.6 \pm 8.5)$
2	$(2/3)(3\text{Pb}(\text{s}) + 2\text{O}_2(\text{g}) = \text{Pb}_3\text{O}_4(\text{s}))$	$\Delta H(2)$	$(2/3) \times -(718.8 \pm 6.3) = -479.2 \pm 4.2$
3	$2\text{Fe}(\text{s}) + (3/2)\text{O}_2(\text{g}) = \text{Fe}_2\text{O}_3(\text{s})$	$\Delta H(3)$	$-(823.4 \pm 3.3)$
4	$2\text{Pb}(\text{s}) + 2\text{Fe}(\text{s}) + (5/2)\text{O}_2(\text{g}) = \text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$	$\Delta H(4)$	$-(1324.2 \pm 11.1)$

TABLE 4

Reaction scheme for the standard molar enthalpy of formation of $\text{PbFe}_5\text{O}_{8.5}(\text{s})$, $\Delta_f H_m^\circ$ at 298.15 K; ΔH denotes enthalpy of reaction $\Delta_f H_m^\circ$ ($\text{PbFe}_5\text{O}_{8.5}, \text{cr}$) = $\Delta H(8)$ = $\Delta H(5)$ + $\Delta H(6)$ + $\Delta H(7)$.

S. no.	Reaction		$\Delta H_m/(\text{kJ}\cdot\text{mol}^{-1})$
1	$(1/3)\text{Pb}_3\text{O}_4(\text{s}) + (5/2)\text{Fe}_2\text{O}_3(\text{s}) = \text{PbFe}_5\text{O}_{8.5}(\text{s}) + (1/6)\text{O}_2(\text{g})$	$\Delta H(5)$	$-(49.7 \pm 8.0)$
2	$(1/3)(3\text{Pb}(\text{s}) + 2\text{O}_2(\text{g}) = \text{Pb}_3\text{O}_4(\text{s}))$	$\Delta H(6)$	$(1/3) \times -(718.8 \pm 6.3) = -239.6 \pm 2.1$
3	$(5/2)(2\text{Fe}(\text{s}) + (3/2)\text{O}_2(\text{g}) = \text{Fe}_2\text{O}_3(\text{s}))$	$\Delta H(7)$	$(5/2) \times -(823.4 \pm 3.3) = -2058.5 \pm 8.3$
4	$\text{Pb}(\text{s}) + 5\text{Fe}(\text{s}) + 4.25\text{O}_2(\text{g}) = \text{PbFe}_5\text{O}_{8.5}(\text{s})$	$\Delta H(8)$	$-(2347.8 \pm 10.7)$

3. Results and discussion

In the compounds $\text{Pb}_2\text{Fe}_2\text{O}_5$ and $\text{PbFe}_5\text{O}_{8.5}$, valences of Pb and Fe are +2 and +3, respectively. Based on the studies on dissolution of Fe_2O_3 under the calorimetric condition within a reasonable time period, 6 M HCl was chosen as calorimetric solvent among the various mineral acids with varying concentrations tested. It also dissolved the ternary compounds, viz., $\text{Pb}_2\text{Fe}_2\text{O}_5$ and $\text{PbFe}_5\text{O}_{8.5}$. However, in the case of PbO, it precipitated as PbCl_2 . On the other hand, Pb_3O_4 was found to dissolve completely in 6 M HCl. This could be probably due to the formation of a soluble complex in conc. HCl facilitated by Pb_3O_4 . Hence, Pb_3O_4 was chosen for determining enthalpy of solution instead of PbO.

The results of the measurements of enthalpy of solution for the compounds $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ and for the starting materials $\text{Pb}_3\text{O}_4(\text{s})$ and $\text{Fe}_2\text{O}_3(\text{s})$ are given in table 2. The thermochemical reaction schemes used to derive the standard molar enthalpies of formation of $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ are given in tables 3 and 4, respectively. As mentioned in table 2, the enthalpy correction for the evolution of O_2 gas was calculated and found to be negligible. However, due to the lack of facility and provision for carrying out controlled dissolution of oxygen in present calorimeter, we could not carry out the measurement of the enthalpy of dissolution of O_2 in 6 M HCl.

The molar enthalpies of solution of $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ and the starting materials $\text{Pb}_3\text{O}_4(\text{s})$ and $\text{Fe}_2\text{O}_3(\text{s})$, deduced from the experiments are $-270.7 \pm 6.3 \text{ kJ}\cdot\text{mol}^{-1}$, $-181.0 \pm 1.4 \text{ kJ}\cdot\text{mol}^{-1}$, $-294.0 \pm 7.3 \text{ kJ}\cdot\text{mol}^{-1}$ and $-53.1 \pm 3.0 \text{ kJ}\cdot\text{mol}^{-1}$, respectively. The enthalpy ΔH (1), of the reaction: $(2/3)\text{Pb}_3\text{O}_4(\text{s}) + \text{Fe}_2\text{O}_3(\text{s}) \rightarrow \text{Pb}_2\text{Fe}_2\text{O}_5(\text{s}) + 0.33\text{O}_2$, in table 3 was obtained by using the Hess's law, by subtracting the enthalpy of dissolution of $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ from that of $(2/3)\text{Pb}_3\text{O}_4(\text{s}) + \text{Fe}_2\text{O}_3(\text{s})$ and is $-21.6 \pm 8.5 \text{ kJ}\cdot\text{mol}^{-1}$. Similarly enthalpy change ΔH (5) of the reaction: $(1/3)\text{Pb}_3\text{O}_4(\text{s}) + (5/2)\text{Fe}_2\text{O}_3(\text{s}) \rightarrow \text{PbFe}_5\text{O}_{8.5}(\text{s}) + 0.16\text{O}_2$ given in table 4, was obtained by subtracting the enthalpy of dissolution of $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ from that of $(1/3)\text{Pb}_3\text{O}_4(\text{s}) + (5/2)\text{Fe}_2\text{O}_3(\text{s})$ and is $-49.7 \pm 8.0 \text{ kJ}\cdot\text{mol}^{-1}$. In all these measurements, the concentration domains are narrow and hence extrapolation at infinite dilution could not be made. As the concentrations are very dilute, in the order of mmol l^{-1} , it is assumed that the enthalpy of solution is constant down to infinite dilution. By using the literature data on standard molar enthalpies of formation of $\text{Pb}_3\text{O}_4(\text{s})$ and $\text{Fe}_2\text{O}_3(\text{s})$ [16], the standard molar enthalpies of formation of $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$ from the constituent elements, were obtained as $-(1324.2 \pm 11.1) \text{ kJ}\cdot\text{mol}^{-1}$ and $-(2347.8 \pm 10.7) \text{ kJ}\cdot\text{mol}^{-1}$, respectively.

4. Conclusions

The standard enthalpy of formation of $\text{Pb}_2\text{Fe}_2\text{O}_5(\text{s})$ and $\text{PbFe}_5\text{O}_{8.5}(\text{s})$, at 298.15 K were determined by acid solution calorimetry and are found to be $-(1324.2 \pm 11.1) \text{ kJ}\cdot\text{mol}^{-1}$ and $-(2347.8 \pm 10.7) \text{ kJ}\cdot\text{mol}^{-1}$, respectively. These thermochemical data are reported for the first time.

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