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The standard molar enthalpies of formation of $\text{Pb}_2\text{CrO}_5(\text{s})$ and $\text{Pb}_5\text{CrO}_8(\text{s})$ by acid solution calorimetry

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ABSTRACT

The standard molar enthalpies of formation, $\Delta_f H_m^\circ$ (298.15 K) of $\text{Pb}_2\text{CrO}_5(\text{s})$ and $\text{Pb}_5\text{CrO}_8(\text{s})$ were determined using an isoperibol calorimeter. The enthalpies of solution of pure compounds, viz., $\text{Pb}_2\text{CrO}_5(\text{s})$, and $\text{Pb}_5\text{CrO}_8(\text{s})$ as well as mixtures viz., $\{\text{PbCrO}_4(\text{s}) + \text{PbO}(\text{s})\}$ and $\{\text{PbCrO}_4(\text{s}) + 4\text{PbO}(\text{s})\}$ in HNO_3 (aq., $8 \text{ mol} \cdot \text{dm}^{-3}$) at $T = 298.15 \text{ K}$ were measured. Using these values, $\Delta_f H_m^\circ$ (298.15 K) of $\text{Pb}_2\text{CrO}_5(\text{s})$ and $\text{Pb}_5\text{CrO}_8(\text{s})$ were determined as $-(1171.5 \pm 1.7) \text{ kJ} \cdot \text{mol}^{-1}$ and $-(1850.6 \pm 8.7) \text{ kJ} \cdot \text{mol}^{-1}$, respectively.

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1. Introduction

A study of the interaction of liquid lead with components of steel in the presence of trace amount of oxygen is needed in the context of lead-cooled nuclear reactors and accelerator driven sub-critical systems (ADSS) [1]. Corrosion in the (lead + steel) systems is mitigated by the formation of an adherent oxide layer on the structural steels. A detailed knowledge of the composition and the thermochemical data of this oxide layer are needed to exploit this process. Understanding the thermochemistry of ternary compounds of the (Pb + Cr + O) and (Pb + Fe + O) systems is of considerable interest in this regard. Preparation and thermal stability of the compounds existing in (Pb + Cr + O) system have been reported in the literature [2–8]. Three stable compounds, viz., $\text{PbCrO}_4(\text{s})$, $\text{Pb}_2\text{CrO}_5(\text{s})$ and $\text{Pb}_5\text{CrO}_8(\text{s})$ exist in this system. The enthalpy of formation of $\text{PbCrO}_4(\text{s})$ at $T = 298 \text{ K}$, $\Delta_f H_m^\circ$, had been determined by Dellien *et al.* [9] by solution calorimetry with NaOH and Na_4EDTA as the solvents. No corresponding data for $\text{Pb}_2\text{CrO}_5(\text{s})$ and $\text{Pb}_5\text{CrO}_8(\text{s})$ are available in the literature. Recently, the partial phase diagram of (Pb + Cr + O) system was established at the author's laboratory by long term equilibration studies [7] and also the standard molar Gibbs free energy of formation of $\text{Pb}_5\text{CrO}_8(\text{s})$, $\text{Pb}_2\text{CrO}_5(\text{s})$, and $\text{PbCrO}_4(\text{s})$ were determined [8] by measuring equilibrium oxygen pressures over appropriate ternary phase fields either by manometry or by solid oxide electrolyte based emf cells. In the present work, the enthalpies of formation of

$\text{Pb}_2\text{CrO}_5(\text{s})$ and $\text{Pb}_5\text{CrO}_8(\text{s})$ at $T = 298 \text{ K}$ have been determined using acid solution calorimetry.

2. Experimental

2.1. Chemicals

High purity PbO (mass fraction purity >0.999 on metal basis, M/s Aldrich Chem. Co., USA) and Cr_2O_3 (mass fraction purity >0.99999 on metal basis, M/s Johnson Matthey Materials Technology, UK) powders were used for preparation of ternary compounds. Thermo gravimetric analyses showed that PbO(s) absorbs carbon dioxide (~0.1 wt%) and $\text{Cr}_2\text{O}_3(\text{s})$ absorbs moisture (~1.8 wt%) on prolonged exposure to ambient air. Hence, PbO(s) and $\text{Cr}_2\text{O}_3(\text{s})$ were calcined for 3 h under flowing argon gas at temperatures of (673 and 973) K, respectively, stored in desiccators and handled with very short exposure to ambient air. Doubled distilled AR grade HNO_3 and HCl which were diluted with the distilled water to the required concentrations were used as the calorimetric solvents.

2.2. Compound preparation

$\text{Pb}_5\text{CrO}_8(\text{s})$, $\text{Pb}_2\text{CrO}_5(\text{s})$ and $\text{PbCrO}_4(\text{s})$ were prepared by solid state reaction between powders of PbO(s) and $\text{Cr}_2\text{O}_3(\text{s})$ taken in the required stoichiometric ratios. The conditions used for preparation of these ternary compounds are described in our earlier publication [7]. The compounds were characterised by X-ray diffraction using a Siemens D500 X-ray powder diffractometer with Cu K α radiation and graphite monochromator. The XRD patterns obtained for $\text{Pb}_5\text{CrO}_8(\text{s})$, $\text{Pb}_2\text{CrO}_5(\text{s})$ and $\text{PbCrO}_4(\text{s})$ matched

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with the patterns reported in JCPDS files for these compounds, viz., 49-0970, 29-0768, and 73-2059, respectively.

2.3. Calorimetric technique

The schematic diagram of the experimental set up is shown in figure 1. The main constituent of the isoperibol calorimeter is the vessel in which the reaction of interest takes place. It was made from a double walled glass Dewar of 0.4 dm³ capacity with an evacuated inter space. The top of the vessel was fitted with an annular Perspex flange having an O-ring groove. It also had a mating top-cover flange with leak tight fittings for introducing a stirrer, a sample bulb, a heater enclosure and a thermistor. The Dewar with all the fittings was kept immersed into a water bath that maintained the temperature within ± 0.002 K so as to minimise the exchange of heat with surroundings. The sample was taken inside a small glass tube provided with a thin walled bulb at the bottom. The glass tube had a glass push rod which was sealed to the inner wall of the sample tube using an O-ring. This arrangement enabled isolation of the sample from surroundings. A stirrer, made up of PTFE, enabled uniform stirring of the calorimetric fluid in the Dewar. The stirrer was driven by a motor so that the frictional heat generated by the stirring was constant both during the experiment as well as during the calibration. The calorimeter was calibrated by electrical as well as by chemical methods. A PTFE coated resistance wire with very low temperature coefficient of resistance (KARMA) was used as a calibration heater such that the change of resistance would be negligible during the calibration. The resistance of the heater was $(4.261 \pm 0.005) \Omega$. The heater was

enclosed in a glass bulb to avoid any possible interaction with the solvent. Silicone oil, which is inert and has a low heat capacity and low vapour pressure, was added to the bulb to immerse the heater. This design enabled conduction of heat from the heater to the calorimetric fluid. The electrical calibration was performed by applying a constant current using a stable power source, for a known period of time. The current and duration of its passage for the electrical calibration was decided by the time taken for the sample to dissolve completely under the experimental conditions and the weight of the sample taken for each experiment. The current was measured across a precision resistor (1.000Ω) of suitable current rating, connected in series. The temperature was measured using a thermistor (M/s PARR Instruments Company, USA) connected to a data acquisition system, interfaced to a personal computer and was recorded as a function of time. Chemical calibration was carried out using KCl (SRM 1655, M/s N.B.S., USA) and double distilled water was used as the solvent. The sample for calibration was weighed accurately and taken inside the sample tube. 0.300 dm³ of double distilled water 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 is in equilibrium with the surrounding water bath, a steady temperature signal was obtained. After a steady signal had been obtained from the control unit, the bulb was broken, the sample was allowed to dissolve in the solvent and the calorimeter signal was recorded. The system was calibrated electrically *in situ* before and after each of this calibration experiment. The temperature change ΔT during

FIGURE 1. Schematics of acid solution calorimeter.

TABLE 1

The enthalpy of solution $\Delta_{\text{sol}}H_m^\circ$ of KCl(s) in 0.300 dm³ of water at $T = 298.15$ K (molar mass of KCl: 74.56).

Run	Mass of KCl/g	Experimental enthalpy change, $\Delta H/J$	Molar enthalpy of solution, $\Delta_{\text{sol}}H_m/(kJ \cdot \text{mol}^{-1})$	Molar enthalpy of solution at infinite dilution, $\Delta_{\text{sol}}H_m^\infty/(kJ \cdot \text{mol}^{-1})$
1	0.2790	65.32	17.46	17.24
2	0.2401	55.99	17.39	17.16
3	0.2782	64.61	17.32	17.08
4	0.2820	65.73	17.38	17.14
5	0.2710	63.18	17.38	17.15
				Average: $(17.15 \pm 0.06)^a$

^a Uncertainty is the standard deviation from the mean.

TABLE 2

The standard molar enthalpies of solution of Pb₂CrO₅(s) and mixture of {PbO(s) + PbCrO₄(s)} in 0.300 dm³ of HNO₃ (aq., 8 mol · dm⁻³) at $T = 298.15$ K.

Sample	Mass of sample/g	Experimental enthalpy change, $\Delta H/J$	Molar enthalpy of solution, $\Delta_{\text{sol}}H_m/(kJ \cdot \text{mol}^{-1})$
Pb ₂ CrO ₅ (s) (M: 546.371 g · mol ⁻¹)	0.2947	-36.18	-67.08
	0.3050	-36.91	-66.12
	0.2648	-31.90	-65.82
	0.2041	-24.45	-65.45
	0.2228	-27.05	-66.33
Average $\Delta_{\text{sol}}H_m = -(66.16 \pm 0.61)^a$			
PbO(s) + PbCrO ₄ (s) (M: 546.371 g · mol ⁻¹)	0.3192	-53.30	-91.23
	0.2861	-48.45	-92.53
	0.3066	-52.24	-93.09
	0.3132	-52.67	-91.88
	0.3635	-62.02	-93.22
	0.3424	-58.59	-93.49
Average $\Delta_{\text{sol}}H_m = -(92.57 \pm 0.87)^a$			

^a Uncertainty is the standard deviation from the mean.

TABLE 3

The standard molar enthalpies of solution of $\text{Pb}_5\text{CrO}_8(\text{s})$ and mixture of $\{4\text{PbO}(\text{s}) + \text{PbCrO}_4(\text{s})\}$ in 0.300 dm^3 of HNO_3 (aq., $8 \text{ mol} \cdot \text{dm}^{-3}$) at $T = 298.15 \text{ K}$.

Sample	Mass of sample /g	Experimental enthalpy change, $\Delta H/\text{J}$	Molar enthalpy of solution, $\Delta_{\text{sol}}H_{\text{m}}/(\text{kJ} \cdot \text{mol}^{-1})$
$\text{Pb}_5\text{CrO}_8(\text{s})$ ($M: 1215.938 \text{ g} \cdot \text{mol}^{-1}$)	0.2518	−73.33	−354.12
	0.2384	−69.76	−355.80
	0.2526	−74.51	−358.66
	0.1959	−58.20	−361.25
	0.2261	−65.29	−351.11
	0.2616	−75.96	−353.06
	Average $\Delta_{\text{sol}}H_{\text{m}} = -(355.67 \pm 3.74)^a$		
$4\text{PbO}(\text{s}) + \text{PbCrO}_4(\text{s})$ ($M: 1215.938 \text{ g} \cdot \text{mol}^{-1}$)	0.2403	−81.74	−413.63
	0.2045	−69.17	−411.30
	0.2135	−72.50	−412.93
	0.2190	−72.02	−399.85
	0.2257	−73.79	−397.53
	Average $\Delta_{\text{sol}}H_{\text{m}} = -(407.05 \pm 7.72)^a$		

^a Uncertainty is the standard deviation from the mean.

TABLE 4

Reaction scheme for deducing the standard molar enthalpy of formation of $\Delta_f H_{\text{m}}^\circ$ at $T = 298.15 \text{ K}$ of $\text{Pb}_2\text{CrO}_5(\text{s})$. $\Delta_f H_{\text{m}}^\circ(\text{Pb}_2\text{CrO}_5, \text{cr}) = \Delta H(4) = \Delta H(1) + \Delta H(2) + \Delta H(3)$.

S. No.	Reaction	$\Delta H_{\text{m}}/(\text{kJ} \cdot \text{mol}^{-1})$
1	$\text{PbO}(\text{s}) + \text{PbCrO}_4(\text{s}) \rightarrow \text{Pb}_2\text{CrO}_5(\text{s})$	$\Delta H(1) \quad -(26.41 \pm 1.06)$
2	$\text{Pb}(\text{s}) + \frac{1}{2} \text{O}_2(\text{g}) \rightarrow \text{PbO}(\text{s})$	$\Delta H(2) \quad -(218.08 \pm 0.07)$
3	$\text{Pb}(\text{s}) + \text{Cr}(\text{s}) + 2\text{O}_2(\text{g}) \rightarrow \text{PbCrO}_4(\text{s})$	$\Delta H(3) \quad -(927.02 \pm 1.39)$
4	$2\text{Pb}(\text{s}) + \text{Cr}(\text{s}) + 2\frac{1}{2}\text{O}_2(\text{g}) \rightarrow \text{Pb}_2\text{CrO}_5(\text{s})$	$\Delta H(4) \quad -(1171.5 \pm 1.7)$

TABLE 5

Reaction scheme for the standard molar enthalpy of formation of $\Delta_f H_{\text{m}}^\circ$ at $T = 298.15 \text{ K}$ of $\text{Pb}_5\text{CrO}_8(\text{s})$. $\Delta_f H_{\text{m}}^\circ(\text{Pb}_5\text{CrO}_8, \text{cr}) = \Delta H(8) = \Delta H(5) + \Delta H(6) + \Delta H(7)$.

S. No.	Reaction	$\Delta H_{\text{m}}/(\text{kJ} \cdot \text{mol}^{-1})$
1	$4\text{PbO}(\text{s}) + \text{PbCrO}_4(\text{s}) \rightarrow \text{Pb}_5\text{CrO}_8(\text{s})$	$\Delta H(5) \quad -(51.38 \pm 8.58)$
2	$4\text{Pb}(\text{s}) + 2\text{O}_2(\text{g}) \rightarrow 4\text{PbO}(\text{s})$	$\Delta H(6) \quad 4 \cdot -(218.08 \pm 0.07)$
3	$\text{Pb}(\text{s}) + \text{Cr}(\text{s}) + 2\text{O}_2(\text{g}) \rightarrow \text{PbCrO}_4(\text{s})$	$\Delta H(7) \quad -(927.02 \pm 1.39)$
4	$5\text{Pb}(\text{s}) + \text{Cr}(\text{s}) + 4\text{O}_2(\text{g}) \rightarrow \text{Pb}_5\text{CrO}_8(\text{s})$	$\Delta H(8) \quad -(1850.6 \pm 8.7)$

electrical calibration as well as during chemical calibration was corrected for heat exchange between calorimeter and surroundings by the method suggested by Kubaschewski and Alcock [10] and was used for the evaluation of the enthalpy change for the reaction.

A similar experimental procedure was adopted for the calorimetric studies involving samples of the lead chromates and PbO and 0.300 dm^3 of HNO_3 (aq., $8 \text{ mol} \cdot \text{dm}^{-3}$) was used as the calorimetric solvent for these samples. The maximum time taken for dissolution for these experiments was around 7 min. The temperature change ΔT during the experiment with Pb – Cr – O compounds was also corrected for heat exchange between calorimeter and surroundings as explained above and was used for the evaluation of the enthalpy change for the reaction. Experiments were repeated to achieve the reproducibility.

3. Results and discussion

The calorimetric results of KCl are given in table 1. Enthalpies of solution correspond to the following reaction:

The experimental values of the enthalpy of solution were used to calculate the enthalpy of solution at infinite dilution. The enthalpy of dilution was estimated by use of the Debye–Hückel limiting equation [11]. The value of $\Delta_{\text{sol}}H_{\text{m}}^\circ$ (298.15 K) for KCl from the experimental results is calculated to be $(17.15 \pm 0.06) \text{ kJ} \cdot \text{mol}^{-1}$. This value agreed well with the value $(17.21 \pm 0.01) \text{ kJ} \cdot \text{mol}^{-1}$ reported by Venugopal *et al.* [12] and the N.B.S. value of $(17.241 \pm 0.018) \text{ kJ} \cdot \text{mol}^{-1}$ [13]. The results obtained for KCl dissolutions established the suitability of the present system for calorimetric measurements.

The results of the measurements of the enthalpy of solution for the compounds $\text{Pb}_2\text{CrO}_5(\text{s})$ and $\text{Pb}_5\text{CrO}_8(\text{s})$ and the mixtures $\{\text{PbCrO}_4(\text{s}) + \text{PbO}(\text{s})\}$ and $\{4\text{PbO}(\text{s}) + \text{PbCrO}_4(\text{s})\}$ are given in tables 2 and 3, respectively. The thermochemical reaction schemes used to derive the standard molar enthalpies of formation of $\text{Pb}_2\text{CrO}_5(\text{s})$ and $\text{Pb}_5\text{CrO}_8(\text{s})$ are given in tables 4 and 5, respectively. The molar enthalpies of solution of $\text{Pb}_2\text{CrO}_5(\text{s})$, $\{\text{PbO}(\text{s}) + \text{PbCrO}_4(\text{s})\}$, $\text{Pb}_5\text{CrO}_8(\text{s})$ and $\{4\text{PbO}(\text{s}) + \text{PbCrO}_4(\text{s})\}$, deduced from the experiments are $-(66.16 \pm 0.61) \text{ kJ} \cdot \text{mol}^{-1}$, $-(92.57 \pm 0.87) \text{ kJ} \cdot \text{mol}^{-1}$, $-(355.67 \pm 3.74) \text{ kJ} \cdot \text{mol}^{-1}$ and $-(407.05 \pm 7.72) \text{ kJ} \cdot \text{mol}^{-1}$, respectively. The enthalpy $\Delta H(1)$, of the reaction: $\text{PbO}(\text{s}) + \text{PbCrO}_4(\text{s}) \rightarrow \text{Pb}_2\text{CrO}_5(\text{s})$, in table 4 was obtained by using the Hess's law, by subtracting the enthalpy of dissolution of $\text{Pb}_2\text{CrO}_5(\text{s})$, from that of $\{\text{PbO}(\text{s}) + \text{PbCrO}_4(\text{s})\}$ and is $-(26.41 \pm 1.06) \text{ kJ} \cdot \text{mol}^{-1}$. Similarly the enthalpy change $\Delta H(5)$ of the reaction: $4\text{PbO}(\text{s}) + \text{PbCrO}_4(\text{s}) \rightarrow \text{Pb}_5\text{CrO}_8(\text{s})$, given in table 5, was obtained by subtracting the enthalpy of dissolution of $\text{Pb}_5\text{CrO}_8(\text{s})$ from that of $\{4\text{PbO}(\text{s}) + \text{PbCrO}_4(\text{s})\}$ and is $-(51.38 \pm 8.58) \text{ kJ} \cdot \text{mol}^{-1}$. The $\Delta_f H_{\text{m}}^\circ$ (298.15 K) of $\text{PbCrO}_4(\text{s})$ was found to be $-927.02 \text{ kJ} \cdot \text{mol}^{-1}$ by Dellien *et al.* [9], the uncertainty in the data was estimated by taking into the account of the error reported in their measurements and the uncertainties given for standard enthalpies of formation of $\text{PbO}(\text{s})$ and $\text{CrO}_3(\text{s})$ in literature [14,15] and is calculated to be $\pm 1.39 \text{ kJ} \cdot \text{mol}^{-1}$. By using the literature data for standard molar enthalpies of formation of $\text{PbO}(\text{s})$ [16] and $\text{PbCrO}_4(\text{s})$ [9], the standard molar enthalpies of formation of $\text{Pb}_2\text{CrO}_5(\text{s})$ and $\text{Pb}_5\text{CrO}_8(\text{s})$ from the constituent elements, were obtained as $-(1171.5 \pm 1.7) \text{ kJ} \cdot \text{mol}^{-1}$ and $-(1850.6 \pm 8.7) \text{ kJ} \cdot \text{mol}^{-1}$, respectively.

4. Conclusion

The standard enthalpies of formation of $\text{Pb}_2\text{CrO}_5(\text{s})$ and $\text{Pb}_5\text{CrO}_8(\text{s})$ at $T = 298.15 \text{ K}$ were measured by acid solution calorimetry and are found to be $-(1171.5 \pm 1.7) \text{ kJ} \cdot \text{mol}^{-1}$ and $-(1850.6 \pm 8.7) \text{ kJ} \cdot \text{mol}^{-1}$, respectively. This is the first report of the data at $T = 298.15 \text{ K}$ for these two compounds.

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