

Thermodynamics of the Pt–Cl system

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Abstract

Using a static method three individual compounds in system of Pt–Cl: PtCl₄, PtCl₃, and PtCl₂ are shown to exist. PtCl was shown not to exist. The enthalpies of formation of platinum chlorides were measured by calorimetry by reduction of the compounds with gaseous hydrogen. The recommended values for the enthalpies of formation at 298.15 K are -137.7 ± 0.3 , 194.2 ± 1.0 , and 245.6 ± 1.9 kJ/mol for PtCl₂(s), PtCl₃(s) and PtCl₄(s), respectively.

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1. Introduction

The existence of three individual condensed platinum chlorides PtCl₂, PtCl₃ and PtCl₄ was established in the investigation of the *P–T–x* diagram of platinum–chlorine system [1–3]. The compound which is assumed by some authors [4–6] to have the composition PtCl is in fact a mixture of compounds described by the general formula Pt₆Cl₁₂·*n*H₂O containing H₂O or [OH[−]] along with platinum and chloride. Thermodynamics of platinum chlorides have been investigated by different methods in a number of works (see Table 1). Only the heat of formation of platinum tetrachloride has been determined by calorimetry [7]. Other authors [1–6,9–11] determined thermodynamic characteristics of platinum chlorides on the basis of the investigation of thermal dissociation of these compounds.

Wohler and Streicher [4] determined the dissociation temperature of platinum chlorides in chlorine flow at a pressure of 1 atm and used the approximate Nernst's equation [8] to calculate heat of dissociation for PtCl, PtCl₂, PtCl₃ and PtCl₄. No convincing evidence for the existence of platinum monochloride was reported therein [4]. Moreover, the analysis of that work gives us grounds to state that chlorine used in the experiments contained moisture and oxygen, which was the reason of the formation of Pt₆Cl₁₂·H_{*n*}O, interpreted by the author as platinum monochlo-

ride. Because of this, we did not take into account the results on PtCl in the calculation of heats of formation of platinum chlorides using Wohler's data (Table 1).

The step corresponding to the formation of PtCl₃ did not occur during the stepwise decomposition of platinum tetrachloride as studied in [5] by means of the isoteniscope procedure. The equilibrium of dissociation $2\text{PtCl}_{3(s)} = 2\text{PtCl}_{2(s)} + \text{Cl}_{2(g)}$ was investigated with specially prepared PtCl₃ samples. In some experiments, after the step corresponding to PtCl₂ dissociation, the authors observed one more step which they attributed to platinum monochloride. They failed to isolate and characterize that product. The standard enthalpies of formation of PtCl₃ and PtCl₂ calculated by us on the basis of experimental data reported in [5] are shown in Table 1.

The results of the investigation of PtCl₂ dissociation process by means of the effusion–torsion procedure were published in [6]. It was stressed that the decomposition of platinum dichloride resulted in the formation of an intermediate compound along with metal platinum and chlorine. This intermediate was assumed to be PtCl. Treating the experimental data on the basis of the second law of thermodynamics the authors determined the heat of platinum dichloride dissociation: $\Delta H^{\circ}_{\text{T}} = 127.356 \pm 0.791$ kJ/mol. This value cannot be considered as reliable, because effusion procedures can give distorted results for platinum chlorides because dissociation equilibrium is a kinetically hindered process.

Krustinson [10] investigated dissociation of platinum dichloride and tetrachloride by means of isoteniscope. These

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Table 1
Heats of formation of platinum chlorides (according to literature data)

Reference	$-\Delta_f H^\circ_{298.15} \text{ kJ/mol}$		
	PtCl ₂ (s)		
	Nernst's equation	Treatment according to second law	Treatment according to third law
[4]	134.60	—	134.98
[5]	—	202.9 ± 18.41	$136.73-140.12$
[6]	—	127.356 ± 0.791	—
[7]	—	—	—
[9]	123	247 ± 121	$124.10-129.20$
[11]	134	—	—

PtCl ₃ (s)			PtCl ₄ (s)		
Nernst's equation	Treatment according to second law	Treatment according to third law	Nernst's equation	Treatment according to second law	Calorimetric data
189.41	—	—	234.51	—	—
—	273.2 ± 25.9	—	—	—	—
—	—	—	—	—	248.5
—	—	—	—	—	—
—	—	—	—	—	—

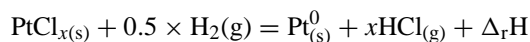
experiments were carried out with better purity, so the decomposition of platinum dichloride proceeded to metal platinum without the intermediate step noticed in [4–6]. The mean value of the heat of PtCl₂ formation calculated by Krustinson using the approximate Nernst's equation and shown in Table 1 is equal to -123 kJ/mol . Data treatment on the basis of the second law of thermodynamics gives the value $-247 \pm 121 \text{ kJ/mol}$. The data of that author on the pressure of PtCl₄ dissociation were not considered by us because the decomposition of platinum tetrachloride proceeded to platinum dichloride leaving aside the step of PtCl₃ formation, that is, following the non-equilibrium route.

Even more unsatisfactory are the data reported in [10], where measurements were carried out under essentially non-equilibrium conditions (under continuous heating of the sample).

Meanwhile, it was stressed as early as in [4,9] that the equilibrium of dissociation of platinum chlorides is established very slowly (within several days). So, the data of [10] cannot be considered reliable.

Shafer and Wiese [11] used the equilibrium quenching method to determine the pressure of PtCl₂ dissociation at 723 K. Then, using the approximate Nernst's equation he found $\Delta_f H^\circ_{298.15} (\text{PtCl}_2) = -134 \text{ kJ/mol}$.

The most reliable data on dissociation pressure of platinum chlorides were obtained using the static method with a membrane null manometer [1–3]. These experiments were carried out in the modes of isothermal and non-isothermal thermostating. The equilibrium pressure of dissociation of platinum chlorides was settled within a very long time (24–200 h and more), so it was possible to approach the equilibrium only from the side of lower pressures. The heats of formation of platinum chlorides, calculated from tensimetric data, required additional verification with an independent procedure because these values could be distorted due to the difficulty of equilibrium establishment and unaccounted features of the behaviour of compounds. We chose calorimetric procedure as such an independent method. Since platinum dichloride and trichloride are almost insoluble in solvents, the methods of solution calorimetry turned out to be unsuitable. We used reduction as the calorimetric reaction:



2. Experimental

2.1. Synthesis and identification of initial compounds

2.1.1. PtCl₄

PtCl₄ was synthesized by thermal decomposition of chloroplatinic acid in chlorine flow ($p_{\text{Cl}_2} = 1 \text{ atm}$) [12] obtained by the interaction of HCl and KMnO₄. The optimal synthesis temperature at which the final product had the composition $n_{\text{Cl}}/n_{\text{Pt}} = 3.94$ and contained a minimal amount of water ($n_{\text{H}_2\text{O}}/n_{\text{Pt}} = 0.01$) was 493 K. Gaseous chlorine was dried preliminarily by passing above H₂SO₄ and CaCl₂. The duration of the reaction was 8–10 h, with the initial weighed portion of 300 mg. To determine platinum and chlorine content of platinum tetrachloride, the weighed portion was reduced in hydrogen flow at 473–573 K,

HCl was absorbed with water; chloride was determined by means of potentiometry. Platinum was determined gravimetrically after calcination in a crucible in the air at 873 K. The synthesized compound was identified by elemental and X-ray phase analyses, as well as by IR spectroscopy. Found (%): Pt, 57.60; Cl, 42.20. PtCl₄—calculated (%): Pt, 57.86; Cl, 42.14. Interplanar spacings for the samples of platinum tetrachloride are satisfactorily described by the data obtained previously [13,14]; the IR spectra coincide with the results reported previously [13,15]. A compound from one and the same lot was examined in calorimetric experiments.

2.1.2. PtCl₃

The method of synthesis of platinum trichloride by the decomposition of PtCl₄ in chlorine flow [12,16] is insufficiently reliable because in many cases it results in the formation of a mixture of PtCl₃ and PtCl₂. A possible reason is essentially non-equilibrium conditions of PtCl₄ decomposition. Because of this, we carried out platinum tetrachloride decomposition in a closed volume. About 8 g of platinum tetrachloride was placed into a three-section ampoule with a volume about 15 ml, the ampoule was pumped out and then sealed. The section with PtCl₄ was placed into a furnace; two other sections were kept at room temperature. The furnace temperature was gradually increased to 483 K; after exposure for 2 days, the gas phase (H₂O) was frozen out and the corresponding section was sealed off. Then temperature was gradually increased during 4 days up to 673–683 K; the substance was kept at this temperature for 2 days. Then the ampoule was put out of the furnace, quickly cooled in air flow, then frozen with liquid nitrogen; the released chlorine was sealed off. The temperature of decomposition of platinum tetrachloride was determined on the basis of tensimetric experiments [1]. The product was identified by means of elemental, X-ray phase analyses, and IR spectroscopy. Found (%): Pt, 64.67; Cl, 35.50. PtCl₃—calculated (%): Pt, 64.72; Cl, 35.28. Interplanar spacings in PtCl₃ coincide with d_{α} values calculated from the structural data [17]. The IR spectrum of PtCl₃ (ν , cm⁻¹): 255 v.s, 302 s, 323 s, 362 m. No data on the IR spectrum of PtCl₃ were found in literature. A compound from one and the same lot was examined in calorimetric experiments.

2.1.3. PtCl₂

Synthesis of β -PtCl₂: according to a known procedure [12] by the decomposition of H₂PtCl₆·6H₂O in chlorine flow at 753 K (fine olive-green powder was formed in the reaction (synthesis 1)).

Synthesis of α -PtCl₂: by means of sublimation of fine platinum dichloride (synthesis 1, weighed portions of 0.5–0.7 g) in a sealed pumped-out ampoule (15 cm³); transfer occurred from the hot region (890 K) into the cool one (800 K). About 2/3 of the initial substance was deposited into the cool region of the ampoule within 10 days. The product was a dark-violet substance composed of dendrites 0.5–1.0 cm in size (synthesis 2).

The synthesized products were identified by means of elemental and X-ray phase analyses, as well as IR spectroscopy. For synthesis 1, found (%): Pt, 73.42; Cl, 26.47. For synthesis 2,

found (%): Pt, 73.26; Cl, 26.67. PtCl₂—calculated(%): Pt, 73.34; Cl, 26.66. Interplanar spacings for the samples obtained by synthesis 1 are in satisfactory agreement with those calculated from the structural data [18]. The interplanar spacings for samples obtained by synthesis 2 coincide with those reported in [17] and do not coincide with those for the samples obtained by synthesis 1. We established previously [19] that the compound obtained in synthesis 1 gets transformed into the compound obtained by synthesis 2 (judging from the set of interplanar spacings) during thermal annealing at 777 K, whereas no reverse transition can be observed. According to Shafer's classification, the products obtained in syntheses 1 and 2 were called β and α modifications of PtCl₂, respectively. For α -PtCl₂, the samples from three lots were used in calorimetric experiments, and for β -PtCl₂ the substances from two lots were used. All the manipulations of packing into ampoules were carried out in a dry box (argon, drying agent: P₂O₅).

2.2. Experimental procedure

In order to determine the enthalpies of formation of platinum chlorides, we measured the heats of reduction of these compounds with gaseous hydrogen. The following major requirements were taken into account when choosing the optimal reduction temperature:

- 1) First of all, it is necessary that the thermodynamic state of the final products of reduction is rather well reproducible in parallel experiments, that is, platinum should be formed in the same structural form and disperse state. Experiments showed that this requirement is met if the substance for use in calorimetric experiments is ground (in a mortar). In this case, platinum is obtained in the form of platinum black with approximately the same grain size distribution for all the three chlorides.
- 2) Reduction should proceed at a sufficient rate. Preliminary investigation of the interaction of platinum chlorides by means of differential thermal analysis and thermogravimetry showed that even in the case of platinum dichloride reduction occurs at a noticeable rate even at 343 K, while the maximal rate corresponds to the temperature of 403 K (with the heating rate of 8° min⁻¹).
- 3) It was necessary to take into account the possibility to carry out calibration experiments with a reference substance. We chose water as the latter; its heat of evaporation is known with a high accuracy [20].

The temperature point that met the listed requirements best of all was 369 K.

Heats of reduction were determined in a flow calorimeter with isothermal shell. The calorimeter was a glass silver-plated Dewar vessel 0.71 in volume, enclosed in a copper shell and immersed into a thermostat filled with silicone oil. The liquid in the calorimeter was mixed at a constant rate.

A weighed portion of the substance in a sealed glass ampoule was placed in the lower part of the glass reaction vessel shaped as a tube with a tap in the lower part and immersed into the

calorimetric liquid by 2/3 of the tube length. Hydrogen flow entered the reaction vessel through a copper heat exchanger located at the thermostat cap and then passed through the tap into a glass spiral which served to increase the pass length of gaseous products inside the calorimeter. The spiral was made of a thin-wall glass tube 5 mm in diameter and about 2 m long; the spiral was completely immersed into the calorimetric liquid. The experiments were carried out with the optimal hydrogen flow rate of 1.8 l/h, at a temperature of 293 K and total pressure of 1 atm.

The ampoule with the substance was broken with a glass pin; the design of the latter allowed us to prevent diffusion of gases from the lower part of the reaction vessel into its upper part.

Temperature of the calorimetric liquid was measured with a thermistor ($r^{369\text{K}} = 11,650 \Omega$) and a direct current bridge with an error of $\pm 0.0001^\circ$, which corresponded to a change in thermistor resistance by 0.03Ω . The indications of the thermistor were read from the chart strip of a plotter. The heat value of the calorimeter was determined by means of calibration over current after each experiment with the substance. The thermal effect and mean temperature of the calibration experiment were set approximately the same as those in the corresponding experiment with the substance. Power conditioner was used as a power supply. Voltage at the calibration heater and current strength in the circuit was measured with a potentiometer. Pulse duration was recorded with a printing chronograph. The working element of the heater was a manganin wire 0.1 mm in diameter having the resistance of 25Ω , which was placed in a thin-wall glass capillary filled with silicone oil. The electric circuit of calibration provided the accuracy of heat measurement with an error not larger than 0.05%. Scattering in heat values did not exceed 0.3% of the mean value. Heat values averaged over the series were used to calculate heats of reaction.

The absence of essential systematic errors of the calorimetric system was established by measuring the heat of water evaporation. The mean result for the heat of evaporation determined in 10 experiments at 369 K was $40.79 \pm 0.13 \text{ kJ/mol}$ (here and below, the error for the 95% confidence interval is shown), which is in good agreement with the literature [20] value 40.8216 kJ/mol .

The cooling constant was 0.0072 min^{-1} as a mean. Deviations from the mean value usually did not exceed $\pm 10\%$.

3. Discussion of results

The results of calorimetric experiments are shown in [Supplementary Table S2](#), where K is the constant of cooling, δ is a correction for heat exchange, Δr is the corrected resistance of thermistor in the calorimetric experiment, τ is the duration of the main period, $\Delta H^\circ_{369,15}$ is the heat effect of the reduction of the corresponding platinum chloride. Enthalpies of reactions were calculated for 298.15 K using thermodynamic values shown in [Table 2](#).

Attention should be paid to a substantial difference between heats of reaction for the ground and non-ground platinum dichloride samples. It should be noted that in the latter case the main period was about two times longer. In addition, a substantial difference was also observed in the appearance of platinum sam-

Table 2

Heat capacity ($C_p^\circ (\text{J mol}^{-1} \text{K}^{-1}) = a + bT + c/T^2$) of crystal platinum, platinum chlorides, gaseous hydrogen and hydrogen chloride within temperature range 298–900 K

Compound	a	b	c
Pt(кр) [21]	28.49	5.27×10^{-3}	0.25×10^5
PtCl ₂ (s) [22]	63.5	21.4×10^{-3}	0.883×10^5
PtCl ₃ (s) [23]	121.34	–	–
PtCl ₄ (s) [23]	146.44	–	–
H ₂ (g) [21]	28.83	–	–
HCl(g) [21]	29.12	–	–

ples. As we have already mentioned above, platinum black was formed as a result of reduction of platinum dichloride ground in a mortar. In the cases when coarse crystals were loaded into calorimetric ampoules, platinum was obtained in the form of light-gray particles conserving the shape of initial crystals. To explain the indicated difference, we assumed that the reduction of coarse crystals results in a less stable state of platinum. Because of this, we accepted the heat of α -PtCl₂ reduction to be equal to the value obtained in experiments with the ground substance. The values of $\Delta_r H^\circ_{298,15}$ for reduction reactions (see [Supplementary Table S2](#)) along with $\Delta_f H^\circ_{298,15}(\text{HCl}) = -92.3121 \pm 0.130 \text{ kJ/mol}$ [24] were used to calculate the enthalpy of formation of platinum chlorides.

When evaluating the reliability of thus obtained heats of formation, the following should be taken into account:

- 1) Reduction resulted in the formation of platinum black, that is, platinum in thermodynamically unstable state.
- 2) Platinum (especially in the form of platinum black) readily sorbs H₂ and HCl. The mentioned circumstances can be taken into account by introducing the corresponding corrections. The latter differ in sign; therefore, they partially compensate each other. To our regret, we failed to estimate the heat of the transition of platinum black into compact platinum and the correction connected with possible sorption of HCl. It may be assumed that both corrections are not large. We measured the heat of H₂ sorption at 369 K experimentally; two parallel experiments gave results -0.63 and -0.88 kJ/g-at. of platinum.

The experiment was carried out as follows. A weighed portion of ground α -PtCl₂ was reduced with hydrogen under the conditions simulating the calorimetric experiment. The resulting platinum black was placed into the calorimetric ampoule; desorption of gases from the surface of platinum was carried out by pumping out (10^{-5} Torr) at 323 K for three hours. After that, the ampoule was sealed under vacuum and placed into the flow calorimeter; the heat released after breaking the ampoule was determined.

Heats of dissociation and heats of formation of platinum chlorides, calculated on the basis of data of calorimetric and tensimetric experiments [1–3], are shown in [Tables 3 and 4](#).

Table 3
Heat of dissociation ($\Delta_r H^\circ_{298.15}$ kJ/mol)

Reaction	Tensimetric	Calorimetric
PtCl ₂ (s) = Pt(s) + Cl ₂ (g)	136.7 ± 0.7 (second law) ^a 137.7 ± 0.3 (third law)	138.6 ± 4.6 (α-PtCl ₂) 138.0 ± 2.4 (β-PtCl ₂)
2PtCl ₃ (s) = 2PtCl ₂ (s) + Cl ₂ (g)	123.1 ± 1.7 (second law)	111.3 ± 5.6
2PtCl ₄ (s) = 2PtCl ₃ (s) + Cl ₂ (g)	104.4 ± 1.8 (second law) 101.70 ± 1.76 (third law)	102.8 ± 2.9

^a Calculation according to the second and third laws of thermodynamics.

Table 4
Heats of formation of platinum chlorides ($-\Delta_f H^\circ_{298.15}$ kJ/mol)

Compound	Tensimetric	Calorimetric	Recommended $-\Delta_f H^\circ_{298.15}$ kJ/mol
PtCl ₂ (s)	137.7 ± 0.3	137.95 ± 2.38 (β-PtCl ₂) 138.62 ± 4.60 (α-PtCl ₂)	137.7 ± 0.3
PtCl ₃ (s)	199.2 ± 1.2	194.2 ± 1.00	194.2 ± 1.0
PtCl ₄ (s)	246.3 ± 1.3	245.6 ± 1.9	245.6 ± 1.9

4. Conclusion

Close values of the heats of formation of platinum dichlorides, calculated from tensimetric measurements and determined by means of calorimetry, provide evidence of the relatively small error connected with the failure of calorimetric experiments to take account of the heats of gas sorption on platinum and the heat of transition of platinum black into the stable form. This error may be assumed to be approximately the same for all platinum chlorides. Because of this, it should be expected that the heats of dissociation of platinum tri- and tetrachloride calculated from calorimetric data would not contain noticeable systematic errors. The enthalpy of platinum dichloride dissociation under standard conditions was calculated on the basis of the second and the third laws of thermodynamics (see Table 3). One can see that both types of treatment give results that are in agreement with each other. Nevertheless, we prefer the results of calculation according to the third law of thermodynamics because the good accuracy of determination of the absolute entropy of PtCl₂(s) achieved earlier [22,25] provided smaller errors in these calculations. Heats of formation of platinum dichloride, calculated from tensimetric and calorimetric data, are in good agreement with each other. However, since the latter gives a larger error of determination, we propose to consider the value calculated from tensimetric data according to the third law of thermodynamics as the recommended value.

A deviation between calorimetric and tensimetric determinations of the enthalpy of PtCl₃(s) formation goes out of the error limits. One of the possible reasons of this deviation can be the estimated thermal capacity values used by us in treating tensimetric data on the dissociation of platinum trichloride. Since the heat of reduction of platinum trichloride, on the basis of which its heat of formation was calculated, has been measured in six calorimetric experiments and exhibited good reproducibility of results, we propose the heat of formation obtained from calorimetric measurements to be considered as the recommended value.

Using the value of the heat of formation of platinum trichloride [3] we calculated the standard heat of formation of PtCl₄ (see Table 4). This value is in good agreement with the results obtained by means of calorimetry, and in satisfactory agreement with the heat of formation of PtCl₄ reported in [7] (−248.5 kJ/mol).

We propose to consider the calorimetric value as the recommended one for the heat of formation of PtCl₄, because when calculating the heat of formation on the basis of tensimetric data we had to rely upon the estimated values of heat capacity for PtCl₄.

The calorimetric investigation carried out by us confirmed the correctness of interpretation of the results of tensimetric experiments [1–3], as well as the reliability of heats of formation calculated on that basis for platinum chlorides (Table 4).

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tca.2008.01.005.

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