## Solid-Phase Reaction of Tetraammineplatinum(II) Chloride with Ammonium Heptamolybdate

E. V. Fesik<sup>*a*,\*</sup>, T. M. Buslaeva<sup>*a*</sup>, T. I. Melnikova<sup>*b*</sup>, and L. S. Tarasova<sup>*c*</sup>

<sup>a</sup> MIREA—Russian Technological University (Lomonosov Institute of Fine Chemical Technologies), Moscow, 119571 Russia

<sup>b</sup> Non-Profit Partnership "Promoting Chemical and Environmental Education," Moscow, 101848 Russia

<sup>c</sup> Krasnoyarsk Scientific Center Federal Research Center, Siberian Branch, Russian Academy of Sciences,

Krasnoyarsk, 660036 Russia \*e-mail: 1707-fesik@mail.ru

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**Abstract**—The solid-phase reaction of  $[Pt(NH_3)_4]Cl_2$  and  $(NH_4)_6Mo_7O_{24}$  under argon in the temperature range from 50 to 500°C was studied by thermal analysis and mass spectrometry. According to the X-ray powder diffraction, X-ray photoelectron spectroscopy, and elemental analysis data, the product consists of ordered Pt<sub>3</sub>Mo phase with an insignificant amount of molybdenum(VI) oxide. The described reaction is accompanied by reduction of the metals, which is promoted by Pt<sup>2+</sup> ions in the presence of ammonia.

Keywords: platinum, molybdenum, thermal analysis

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Two- and multi-component solid-phase syntheses of materials possessing new physical, chemical, and functional properties have attracted much attention [1–11], and chemical methods of synthesis of powders with different compositions are mainly discussed. Materials based on the platinum–molybdenum system [4–11] are used as supported catalysts in the oxidation [4] and hydrogenation of carbon monoxide [5], dehydrogentation of hydrocarbons [6, 7], electrochemical oxidation of methanol in an alkaline electrolyte [8, 9], reforming [10], and cracking [11].

Platinum–molybdenum bimetallic powders are commonly prepared by thermal decomposition of platinum and molybdenum compounds and/or their mixtures after joint or successive impregnation of an appropriate porous support (carbon, oxide, ceramic, etc.), followed by drying and calcination in a reducing atmosphere [4–11]. Usually, H<sub>2</sub>[PtCl<sub>6</sub>]·6H<sub>2</sub>O, [Pt(NH<sub>3</sub>)<sub>4</sub>] Cl<sub>2</sub>·H<sub>2</sub>O, (NH<sub>4</sub>)<sub>6</sub>Mo<sub>7</sub>O<sub>24</sub>·4H<sub>2</sub>O, Na<sub>2</sub>MoO<sub>4</sub>·2H<sub>2</sub>O, and MoO<sub>2</sub>(C<sub>5</sub>H<sub>7</sub>O<sub>2</sub>)<sub>2</sub> are used as precursors to platinum– molybdenum systems

The solid-phase reactions of tetraammineplatinum(II) chloride with ammonium chromate and dichromate in an inert atmosphere in the temperature range from 50 to 500°C give  $Pt_{1-x}Cr_x$  metal solid solution [a = 3.9271(6) Å]

and ordered  $Pt_xCr_y$  phase, respectively [12]. In the reaction with ammonium perrhenate under similar conditions, no reduction to rhenium metal is observed, and a mixture of Pt and ReO<sub>2</sub> is formed [13]. These results are very consistent with the data of [3] where CuPt, NiPt, CoPt, and other bimetallic alloys were prepared by adding a mixture of metal salts to a solution of octadecylamine at 180°C.

We have studied the solid-phase reaction of tetraammineplatinum(II) chloride with ammonium heptamolybdate under argon in the temperature range from 50 to 500°C with the goal of obtaining fine platinum-molybdenum bimetallic powders. According to the thermogravimetric analysis data, the reaction includes five steps with six endothermic effects. The low-temperature endothermic effect with its maximum at 95.5°C is accompanied by an insignificant weight loss (1.3%,  $\Delta m_{\text{theor}} =$ 1.35%). It corresponds to elimination of two water and two ammonia molecules, which were detected in the gaseous phase by mass spectrometry. In the temperature range 200-300°C, the DSC curve showed a broadened double endothermic peak at 265.2/278.3°C, which is likely to arise from decomposition of [Pt(NH<sub>3</sub>)<sub>4</sub>]Cl<sub>2</sub> via removal of two ammonia molecules with the formation

	Temperature range, °C	Temperature, °C		Weight loss $\Delta m$ , %			Compound	
Stage no.		DTG	DSC	experimental	calculated	<i>m</i> (compound)	gas phase	solid phase
1	50-200	92.4	95.5	1.3	1.35	17 (NH <sub>3</sub> ) 18 (H <sub>2</sub> O)	2NH <sub>3</sub> 2H <sub>2</sub> O	Pt <sub>12</sub> Mo <sub>7</sub> O <sub>22</sub> Cl <sub>24</sub> N <sub>52</sub> H <sub>158</sub>
2	200–300	229.2 262.5 277.3	228.8 265.2 278.3	11.2	11.20	17 (NH <sub>3</sub> ) 18 (H <sub>2</sub> O)	32NH <sub>3</sub> 2H <sub>2</sub> O	$Pt_{12}Mo_7O_{20}Cl_{24}N_{20}H_{58}$
		$\Sigma \Delta m_{1-2}$		12.5	12.55			
3	300–350	335.8	337.9	20.2	20.00	17 (NH <sub>3</sub> ) 18 (H <sub>2</sub> O) 35 (Cl) 28 (N <sub>2</sub> )	9NH <sub>3</sub> 5H <sub>2</sub> O 20Cl 3N <sub>2</sub>	Pt <sub>12</sub> Mo <sub>7</sub> O <sub>15</sub> Cl <sub>4</sub> N <sub>5</sub> H <sub>21</sub>
		$\Sigma \Delta m_{2-3}$		32.7	32.55	× 2)	2	
4	350–500	364.3	356.2 366.9 500.0	6.6	6.36	17 (NH <sub>3</sub> ) 18 (H <sub>2</sub> O) 35 (Cl) 28(N <sub>2</sub> )	3NH <sub>3</sub> 6H <sub>2</sub> O 4Cl N <sub>2</sub>	Pt <sub>12</sub> Mo <sub>7</sub> O <sub>9</sub>
		$\Sigma \Delta m_{3-4}$		39.3	38.9			
		<i>m</i> (solid residue)		60.7	61.10			

**Table 1.** Combined thermal and mass spectrometric analysis of a mixture of  $[Pt(NH_3)_4]Cl_2$  and  $(NH_4)_6Mo_7O_{24}$  and assumed compositions of solid intermediate products

of *trans*-[Pt(NH<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>] [17]. The left branch of that peak is unsymmetrical, which suggests simultaneous dehydration and decomposition of  $(NH_4)_6Mo_7O_{24}$  according to Eqs. (1) and (2) (DSC maximum at 228°C).

$$(\mathrm{NH}_{4})_{6}\mathrm{Mo}_{7}\mathrm{O}_{24}\cdot4\mathrm{H}_{2}\mathrm{O} \rightarrow (\mathrm{NH}_{4})_{4}\mathrm{H}_{2}\mathrm{Mo}_{7}\mathrm{O}_{24}\cdot1.5\mathrm{H}_{2}\mathrm{O} + 2.5\mathrm{H}_{2}\mathrm{O}\uparrow + 2\mathrm{NH}_{3}\uparrow, \qquad (1)$$

$$\begin{split} \mathrm{NH}_{4}_{4}\mathrm{H}_{2}\mathrm{Mo}_{7}\mathrm{O}_{24} \cdot 1.5\mathrm{H}_{2}\mathrm{O} &\rightarrow (\mathrm{NH}_{4})_{3}\mathrm{H}_{3}\mathrm{Mo}_{7}\mathrm{O}_{24} \cdot 0.5\mathrm{H}_{2}\mathrm{O} \\ &+ \mathrm{H}_{2}\mathrm{O}\uparrow + \mathrm{NH}_{3}\uparrow. \end{split} \tag{2}$$

The gas phase contained ammonia (*m*/*z* 17) and water H<sub>2</sub>O (*m*/*z* 18) with their maximum release at 265.6 and 276.9°C, as well as NO (*m*/*z* 30) with its maximum at 265°C. The latter is formed by oxidation of ammonia. The TG curve showed a significant weight loss in the temperature range 200–290°C (11.2%,  $\Delta m_{\text{theor}} = 11.2\%$ ) due to elimination of 32NH<sub>3</sub> and 2H<sub>2</sub>O (Table 1). The overall weight loss in the first and second stages was 12.5% ( $\Delta m_{\text{theor}} = 12.55\%$ ), and it was accompanied by absorption of heat ( $\Delta H = 333.5 \pm 6$  J/g).

The X-ray powder diffraction (XPD) analysis of intermediate compound obtained at 278°C showed the presence of two phases, *trans*-[Pt(NH<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>] and

a platinum-based cubic solid solution  $Pt_{1-x}Mo_x$  [space group *Fm-3m*, a = 3.935(7) Å].<sup>1</sup> As in the systems derived from tetraammineplatinum(II) chloride and ammonium chromate, dichromate, or ammonium perrhenate [12, 13], the  $Pt_{1-x}Mo_x$  solid solution is likely to be formed as a result of reduction of  $Mo_7O_{24}^{6-}$  to molybdenum metal in the presence of  $Pt^{2+}$  and ammonia.

The DSC curve showed a strong endothermic effect in the temperature range 300–350°C with its maximum at 337.9°C. Intense decomposition of the components was accompanied a weight loss of 20.2% ( $\Delta m_{\text{teor}} = 20.0\%$ ), which was twice as high as that in the preceding stages; it corresponded to removal of 20Cl, 5H<sub>2</sub>O, 9NH<sub>3</sub>, and 3N<sub>2</sub> (Table 1). According to the XPD analysis data, the thermolysis products formed at 340 and 367°C are Pt<sub>1-x</sub>Mo<sub>x</sub> cubic solid solutions [space group *Fm*-3*m*, a = 3.930(5) and 3.930(2) Å] and a small amount of molybdenum oxides.

<sup>&</sup>lt;sup>1</sup> We failed to identify phase composition of the intermediate product obtained at 265°C.

State	Peak	$E_{\rm b}$ , eV	Fraction on the surface, mol %						
Pt <sup>0</sup>	$Pt4f_{7/2}$	71.7	8.70						
Pt(OH) <sub>2ads</sub>	$Pt4f_{7/2}$	72.8	1.90						
PtO/Pt <sup>2+</sup>	$Pt4f_{7/2}$	73.8	1.01						
MoO <sub>2</sub>	$Mo3d_{5/2}$	229.5	0.90						
Mo <sup>5+</sup>	$Mo3d_{5/2}$	230.9	1.72						
MoO <sub>2</sub> /Mo <sup>5+</sup>	$Mo3d_{5/2}$	232.1	2.10						
[MoO <sub>4</sub> ] <sup>2-</sup> /Mo <sup>5+</sup>	$Mo3d_{5/2}$	232.9	9.50						
MoO <sub>3</sub> /Mo <sup>6+</sup>	$Mo3d_{5/2}$	234.0	9.40						
Oxides	O1s	530.6	33.60						
Oxides/OH <sub>ads</sub>	O1s	531.9	25.90						
H <sub>2</sub> O <sub>ads</sub>	O1 <i>s</i>	533.3	5.20						

**Table 2.** Electronic states, fraction of substances on the sample surface, and binding energies

The final decomposition stage in the temperature range 350-500°C gave two weakly separated endothermic peaks at 356 and 366.9°C on the DSC curve. The gas phase contained atomic chlorine, ammonia, water, and nitrogen, indicating that the reduction to form metallic phases continued. It is important that the mass spectra of the gas phase at 338 and 365°C showed a peak of atomic chlorine (m/z 35) in addition to molecular nitrogen (m/z 35)28). The presence of atomic chlorine is likely to indicate not only complete decomposition of *trans*- $[Pt(NH_3)_2Cl_2]$ but also a probable contribution of chloride ion as reducing agent. The weight loss (6.6% of the initial weight,  $\Delta m_{\text{theor}} = 6.36\%$ ) corresponds to removal of 4Cl,  $3NH_3$ ,  $6H_2O$ , and  $N_2$ . The overall heat effect for  $T_3 =$ 337.9,  $T_4 = 356.2$ , and  $T_5 = 366.9$ °C is  $\Delta H = 325 \pm 3$  J/g. The formation of the final solid product was complete at 400°C, and the weight of the solid residue almost did not change in the temperature range from 400 to 500°C. The overall weight loss was 39.3% ( $\Delta m_{\text{theor}} = 38.9\%$ ), the weight of the solid residue was 60.7% ( $\Delta m_{\text{theor}} = 61.09\%$ ), and the metal content of the mixture was 55.2%. The weight of the solid residue based on 12Pt and 7MoO<sub>3</sub> was 64.7% or 61.1% based on 12Pt, 4Mo, and 3MoO<sub>3</sub>. In our opinion, the latter seems more pertinent (Table 1). The fact that the experimental weight loss is insignificantly higher than the calculated value suggests the possibility of more complete reduction of  $Mo_7O_{24}^{6-}$  to the metal. It should be emphasized that, according to the XPD data, the product obtained at 500°C (which was already formed in the final state at 400°C) contained platinum metal and ordered Pt<sub>3</sub>Mo intermetallics.

The presence of an ordered  $Pt_3Mo$  phase in the platinum–molybdenum binary alloy ( $x_{Pt} > 0.5$ ) at 773 K

was reported previously [18]. The XPD patterns showed a peak at  $2\theta = 42^{\circ}-43^{\circ}$  indicates formation of various intermetallic Pt<sub>x</sub>Mo<sub>y</sub> phases [19–21]. The elemental composition of the compound obtained at 500°C under argon corresponds to the formula Pt<sub>0.72</sub>Mo<sub>0.21</sub>O<sub>0.07</sub>. An insignificant amount of oxygen therein may be related to the presence of traces of molybdenum oxide phases which were not included in the solid solution.

The electronic states and elemental composition (Table 2) were determined by X-ray photoelectron spectroscopy (XPS). The Pt4*f* and Mo3*d* spectra are represented by the spin–orbit doublets Pt4 $f_{7/2}$ –Pt4 $f_{5/2}$  and Mo3 $d_{5/2}$ –Mo3 $d_{3/2}$ , respectively. Each spectrum consisted of several components corresponding to different electronic states of the elements. According to the XPS data, the ratio Pt/Mo/O is 11.6:23.6:64 mol %; this suggests that a part of molybdenum not included in the solid solution undergoes oxidation. Line shape analysis revealed platinum metal on the surface ( $E_{\rm b}(\text{Pt4}f_{7/2}) = 71.7 \text{ eV}$ ) in an amount of 8.7%, as well as several oxidized platinum states such as Pt(OH)<sub>2</sub> and PtO (1.9 and 1%, respectively).

Molybdenum is present in the surface layer in different forms, in particular MoO<sub>3</sub> [9.4 mol %,  $E_b(Mo3d_{5/2}) =$ 234 eV], Mo<sup>5+</sup> (13.3 mol %), and MoO<sub>2</sub> (0.9 mol %). Presumably, during the process molybdenum is partially reduced to form a solid solution with platinum. That part of molybdenum which was not included in the solid solution, remains in the oxidized state, which is well consistent with the XPD and TG data.

The observed peak of oxygen  $O_{1s}$  has a complex structure and consists of several components:  $E_b(O1s) = 530.6, 531.9$ , and 533.3 eV. The first component arises from oxide oxygen, and the second and third, to hydroxy group and water, respectively; the latter could appear on the sample surface as a result adsorption of water from air during sample preparation for the analysis.

Our results led us to presume that  $[Pt(NH_3)_4]Cl_2$  and  $(NH_4)_6Mo_7O_{24}$  react under argon according to Eq. (3); the fraction of molybdenum oxide phase in the thermolysis product is much lower than the fraction of metal phase.

 $12[Pt(NH_3)_4]Cl_2 + (NH_4)_6Mo_7O_{24} \rightarrow 12Pt + 4Mo + 3MoO_3$  $+ 4N_2\uparrow + 46NH_3\uparrow + 15H_2O\uparrow + 12Cl_2\uparrow.$ (3)

As found by XPD analysis, the product obtained from the same salts under oxic conditions (air) at 500°C consisted of two phases, platinum-based cubic solid solution [Pt<sub>1-x</sub>Mo<sub>x</sub>, space group *Fm*-3*m*, *a* = 3.928(3) Å] and oxide  $MoO_3$ ; the composition of that product matches the formula  $Pt_{0.68}Mo_{0.19}O_{0.13}$ .

The reaction of  $[Pt(NH_3)_4]Cl_2$  with  $(NH_4)_6Mo_7O_{24}$  in aqueous alkali at 190°C (under pressure) gave a product consisting of platinum metal phase and platinum-based cubic solid solution  $Pt_{0.734}Mo_{0.266}$ , a = 3.935(4) Å [22]. This reaction is described by Eq. (4).

$$6[Pt(NH_3)_4]Cl_2 + (NH_4)_6Mo_7O_{24} + 12KOH \rightarrow 6Pt^0 + 7Mo^0 + 9N_2 + 12NH_3 + 36H_2O + 12KCl.$$
(4)

Unlike compounds formed in the solid-phase reactions (argon, air) in the temperature range from 50 to 500°C, no oxide phases were detected in the products obtained under pressure.

Thus, the solid-phase reaction of  $[Pt(NH_3)_4]Cl_2$  and  $(NH_4)_6Mo_7O_{24}$  at 500°C in an inert atmosphere (argon) or on exposure to air gives molybdenum and platinum metal phases, which confirms our assumption that  $Pt^{2+}$  ions in the presence of ammonia promote reduction of  $Mo_7O_{24}^{6-}$  to molybdenum metal. Our results indicate the possibility of obtaining finely dispersed platinum–molybdenum intermetallic powders.

## **EXPERIMENTAL**

Analytical grade ammonium heptamolybdate tetrahydrate  $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$  (GOST 3765-78, REAKHIM) was commercial product; tetraammineplatinum(II) chloride monohydrate  $[Pt(NH_3)_4]Cl_2\cdot H_2O$ was prepared according to [14] from chloroplatinic acid (TU 2612-059-00196533-2002, Krastsvetmet). The other reagewnts used were of chemically pure or higher grade.

Thermogravimetric analysis was performed on a Netzsch STA 449S Jupiter instrument (Germany) coupled with a QMS 403 C Aeolos mass spectrometer (Krasnoyarsk Regional Joint Center, Federal Research Center "Krasnoyarsk Scientific Center," Siberian Branch, Russian Academy of Sciences). A minimum amount of water was added to a mixture of required amounts of the initial compounds. The mixture was thoroughly stirred in an evaporating dish which was placed in a drying oven and kept at 100°C until constant weight. A sample of the solid mixture was transferred to a corundum crucible and heated at a rate of 10 deg/min in a stream of argon (40 mL/min).<sup>2</sup> Intermediate products of thermal decomposition were obtained (for phase and chemical analysis) in a flow quartz reactor placed in a tubular furnace in a stream of argon (40 mL/min) at a temperature of the corresponding endo- or exothermic peak on the TG curve.

The X-ray powder diffraction patterns were recorded on a Thermo Fisher Scientific ARL X'TRA instrument (Switzerland) with monochromatized  $CuK_{\alpha}$  radiation  $(\lambda = 1.54056 \text{ Å})$ ; linear wavelength correction (1.54433 Å), scanning with a step of 2°; pulse duration 3 s; 2 $\theta$  range 5°–80°. Phases were identified using PCPDFWIN PDF2 automated database. The unit cell parameters were refined by DICVOL04 program [15]. The X-ray photoelectron spectra of powder samples were measured with an Omicron ESCA+ spectrometer; monochromatized Al radiation with an energy of 1486.6 eV and a power of 252 W; analyzer chamber pressure  $\leq 10^{-9}$  torr; analyzer energy 20 eV. The positions of lines of elements in the surface layer were referenced to the C1s peak of residual hydrocarbon impurities in the preparation of powders and from atmosphere. The elemental compositions of the thermolysis products were determined by energy dispersive spectroscopy using a Jeol JED-2300 Analysis Station (Japan); accelerating voltage 20 kV.

## CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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 $<sup>^{2}</sup>$  The reactions on exposure to air were carried out in a tubular furnace on heating at a rate of 10 deg/min.

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