ISSN 1070-3284, Russian Journal of Coordination Chemistry, 2024, Vol. 50, No. 3, pp. 211–216. © Pleiades Publishing, Ltd., 2024.

# Dimolybdenum Perfluorotetrabenzoate and Silver Perfluorocyclohexanoate: Synthesis, Evaporation, and Thermodynamic Characteristics

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Received May 26, 2023; revised September 12, 2023; accepted September 18, 2023

**Abstract**—Anhydrous dimolybdenum perfluorotetrabenzoate $Mo_2(OOCC_6F_5)_4$ (**I**) and silver perfluorocyclohexanoate $AgOOCC_6F_{11}$ (**II**) are synthesized for the first time. Complex **I** is synthesized by the transcarboxylation of dimolybdenum tetraacetate with pentafluorobenzoic acid. Compound **II** is synthesized from freshly prepared silver oxide and perfluorocyclohexanoic acid. The evaporation of the complexes is studied by the Knudsen method with mass spectral analysis of the gas phase. The sublimation of $Mo_2(OOCC_6F_5)_4$ is congruent. The enthalpy of sublimation and the equation of the temperature dependence of the vapor pressure are found. The evaporation of $AgOOCC_6F_{11}$ is accompanied by the complete thermal decomposition with the formation of Ag(s) and mainly $C_6F_{12}$, $C_6F_{10}$, and $CO_2$ molecules. The standard enthalpies of thermal decomposition ($\Delta_r H^\circ_{298.15}(5) = 439.5 \pm 16.4$ kJ/mol, $\Delta_r H^\circ_{298.15}(6) = 325.2 \pm 14.0$ kJ/mol) and formation of the silver complex ($\Delta_f H^\circ_{298.15}$ ($AgOOCC_6F_{11}$, c) = $-2751.0 \pm 24.4$ kJ/mol) are determined.

**Keywords:** synthesis, dimolybdenum perfluorotetrabenzoate, silver perfluorocyclohexanoate, mass spectrometry, standard enthalpy of formation, evaporation

**DOI:** 10.1134/S1070328423601310

## INTRODUCTION

The evaporation of many carboxylic acid salts is congruent with the transition of the substance to the gas phase in the form of monomeric, dimeric, trimeric, and other molecules [1–7]. A similar evaporation mechanism makes it possible to consider these compounds as promising precursors in the CVD procedure for the preparation of various functional materials as films and coatings. Nowadays almost the single known exception is the evaporation of perfluorobenzoic acid salts accompanied by the transition to the gas phase of metal perfluorodiphenyl molecules for monovalent metals and metal perfluoropolyphenyl molecules for polyvalent metals [8, 9], for instance,

$$2AgOOCC_6F_5(s) = 2Ag(s) + 2CO_2(g) + (C_6F_5)_2(g), \quad (1)$$

$$Zn(OOCC_6F_5)_2(s) = Zn(C_6F_5)_2(g) + 2CO_2(g), \quad (2)$$

$$In(OOCC_6F_5)_3(s) = In(C_6F_5)_3(g) + 3CO_2(g). \quad (3)$$

Therefore, it seems very interesting to study the evaporation of dimolybdenum perfluorotetrabenzoate (**I**), whose carboxylate compounds are congruently sublimed [10–14] with the transition to the gas phase as dimeric molecules with a very strong M—M quaternary bond [11, 12]. Many molybdenum carboxylate compounds with a complicated structure of the ligand are rather resistant to air oxygen [10], which significantly facilitates their storage and use in technologies of production of heat-resistance metallic and carbide coatings. Interest in studying the thermodynamic characteristics of silver perfluorocyclohexanoate (**II**) is due to a possibility of using **II** in the synthesis and determination of the standard enthalpies of formation of perfluorocyclohexanoic acid salts by the previously proposed procedure based on the sublimation of a heterophase mixture of silver carboxylate and metal [15].

This work is devoted to the synthesis and study of the evaporation of complexes $Mo_2(OOCC_6F_5)_4$ (**I**) and $AgOOCC_6F_{11}$ (**II**).

## EXPERIMENTAL

Anhydrous dimolybdenum perfluorotetrabenzoate (**I**) was synthesized by the transcarboxylation of molybdenum acetate with pentafluorobenzoic acid in anhydrous acetonitrile without air oxygen and moisture access. The product was isolated in an anhydrous medium using the standard Schlenk technique and a vacuum line. Acetonitrile (reagent grade, Khimmed) was dried over phosphorus(V) oxide and stored over activated molecular sieves (4 Å). Hexane (reagent grade, Khimmed) was dehydrated over metallic sodium and stored over sodium mirror. Tetrahydrofuran was distilled over sodium hydroxide, dehydrated with metallic sodium, and stored over the sodium complex with benzophenone. The dried solvents were sampled by condensation prior to synthesis. Molybdenum acetate (98%, Aldrich) and pentafluorobenzoic acid (reagent grade, Khimmed) were used without additional purification.

The IR spectra of all compounds were recorded in a range of 400–4000 $cm^{-1}$ on a Perkin Elmer Spectrum 65 spectrophotometer equipped with a Quest ATR Accessory instrument (Specac) by the attenuated total internal reflectance (ATR) method. Elemental analyses of all compounds were conducted on a EuroEA-3000 C,H,N,S automated analyzer (EuroVektor). X-ray fluorescence microprobe analysis was carried out on a Bruker M4 Tornado spectrometer.

**Synthesis of $Mo_2(OOCC_6F_5)_4$ (I).** Weighed samples of molybdenum acetate (0.426 g, 1.00 mmol) and perfluorobenzoic acid (0.932 g, 4.4 mmol) were placed in a glass ampule and degassed in a dynamic vacuum for 20 min on cooling the ampule walls with liquid nitrogen. Acetonitrile (20 mL) was condensed into the ampule. The reaction mixture was heated in an oily bath at 100°C and stirred to the complete dissolution of the reagents (1 h) and the appearance of the dark brown color of the reaction mixture. Then acetonitrile was removed by condensation. The formed brown powder was heated in a dynamic vacuum in an oily bath at 150°C to remove acetic acid and pentafluorobenzoic acid excess. Then the product was washed with anhydrous hexane (20 mL), the formed yellow solution was filtered (Schott filter, G4), hexane was removed on heating with the formation of a yellow finely crystalline precipitate, and the last step was repeated five times. The yield was 0.77 g (74%). For $C_{28}F_{20}O_8Mo_2$ anal. calcd.: C, 32.46%; found: C, 32.31%.

IR (ATR; ν, $cm^{-1}$): 1654 w, 1525 vs, 1488 s, 1428 s, 1390 vs, 1290 m, 1115 m, 992 vs, 942 m, 817 w, 754 s, 679 m, 585 w, 519 m, 443 m.

Anhydrous $AgOOCC_6F_{11}$ (**II**) was synthesized from freshly prepared silver oxide and perfluorocyclohexanoic acid. All manipulations on the synthesis of the initial silver oxide and target product were carried out in degassed distilled water without air access using a vacuum line. Silver nitrate (reagent grade, ABCR) and perfluorocyclohexanoic acid (95%, PiM-Invest) were used as received.

**Synthesis of $AgOOCC_6F_{11}$ (II).** A solution of perfluorocyclohexanoic acid (1.372 g, 4.0 mmol) in distilled water (10 mL) was added to $Ag_2O$ freshly prepared from $AgNO_3$ (0.720 g, 4.0 mmol) and NaOH (0.160 g, 4.0 mmol). The dissolution of $Ag_2O$ and formation of a colorless transparent solution were observed. The solution was filtered on the Schott filter (G4). The filtrate was evaporated in a dynamic vacuum, and the formed white powder was dried in an oily bath (100°C, 20 h) under reduced pressure. The yield was 1.640 g (95%). For $C_7F_{11}O_2Ag$ anal. calcd.: C, 19.42%; found: C, 19.50%.

IR (ATR; ν, $cm^{-1}$): 1628 vs, 1390 s, 1314 s, 1262 m, 1216 vs, 1174 vs, 1138 s, 1056 w, 1018 s, 964 vs, 908 s, 872 w, 766 s, 713 w, 682 w, 633 s, 588 w, 524 m, 462 m, 426 w.

The thermodynamics of the evaporation of the synthesized molybdenum and silver complexes was studied by the Knudsen effusion method with mass spectral analysis of the gas phase. Standard molybdenum effusion chambers with the ratio of the evaporation surface area to the effusion surface area $S_{vap}/S_{eff} \approx 600$ were used, and the temperature was measured with a Pt–Pt/Rh thermocouple and maintained constant with an accuracy of ±1 K.

## RESULTS AND DISCUSSION

The mass spectrum of the gas phase over $Mo_2(OOCC_6F_5)_4$ (**I**) is given in Table 1. The form of the mass spectrum allows one to draw only one conclusion: the $Mo_2(OOCC_6F_5)_4$ molecules exist over the molybdenum complex in the gas phase. Any conclusions about the qualitative composition cannot be made from the study of the energy of saturated vapor ionization because of the low intensity of the molecular ion.

The experiments on the isothermal sublimation of the known weighed sample (4–5 mg) of the molybdenum complex were conducted to establish the character of evaporation and gas phase composition and to calculate the absolute partial pressures (Fig. 1). Since the intensities of all ion currents are constant during the evaporation of the weighed sample, their changes during "burning out" of the complex are symbate, and no nonvolatile residue is observed in the effusion chamber, we can conclude that the sublimation of dimolybdenum perfluorotetrabenzoate is congruent and the saturated vapor consists of only one molecular type: $Mo_2(OOCC_6F_5)_4$. The experiments on complete isothermal sublimation made it possible to calculate the absolute partial pressure of the $Mo_2(OOCC_6F_5)_4$ molecules at the temperatures $T_1 = 530$ K and $T_2 = 508$ K ($p_1 = 0.37$ Pa and $p_2 = 0.068$ Pa) by the Hertz–Knudsen equation.

The standard enthalpy of sublimation of complex **I** was calculated by least squares using the Clausius–Clapeyron equation and the temperature dependences of the intensities of the main ion currents in the mass

**Table 1.** Mass spectrum of $Mo_2(OOCC_6F_5)_4$ at $T = 530$ K ($U_{ioniz} = 60$ V)*

| $m/z$ | Ion | Intensity, rel. units | $m/z$ | Ion | Intensity, rel. units |
|---|---|---|---|---|---|
| 86 | $C_4F_2^+$ | 0.5 | 378 | $C_6F_5PFB^{+}$** | 0.6 |
| 112 | $C_3F_4^+$ | 11 | 526 | $Mo_2(C_6F_5)_2^+$ | 0.6 |
| 144 | $C_5F_3CO^+$ | 1.7 | 564 | $Mo_2F_2(C_6F_5)_2^+$ | 0.6 |
| 167 | $C_6F_5^+$ | 21 | 614 | $Mo_2(PFB)_2^+$ | 1.5 |
| 195 | $C_6F_5CO^+$ | 100 | 825 | $Mo_2(PFB)_3^+$ | 2.2 |
| 260 | $C_6F_5COCF_3^+$ | 0.7 | 869 | $Mo_2(PFB)_4–C_6F_5^+$ | 1.0 |
| 352 | $C_6F_5OC_6F_5^+$ | 0.1 | 1036 | $Mo_2(PFB)_4^+$ | 0.1 |

* The isotopic composition of Mo was taken into account in the mass spectrum.
** PFB = $OOCC_6F_5$.

spectrum of the saturated vapor (Table 2). A sufficiently large weighed sample of the substance (80–100 mg) was used when recording the temperature dependences to have the substance remained in the cell after the end of the experiment. The substance was unchanged after partial sublimation; that is, no melting occurred in the studied temperature range. Note that the equal enthalpies found for different ions confirm our conclusion about the monomolecular composition of the saturated vapor above the molybdenum complex.

The determined thermodynamic characteristics of the evaporation made it possible to present the temperature dependence of the saturated vapor pressure (Pa) of the $Mo_2(OOCC_6F_5)_4$ molecules in a temperature range of 448–536 K as follows:

$$\log p = \frac{(-9980 \pm 1050)}{T} + (18.4 \pm 0.1). \quad (4)$$

One of the most interesting and unexpected results of this part of the work is related to the surprising mass spectrum of the $Mo_2(OOCC_6F_5)_4$ molecule. As can be seen from Table 1, the main ion in the mass spectrum of this molecule is the fragment ion of the $OOCC_6F_5$ ligand. A similar dissociative ionization of the salt is observed for the first time. As a rule, this process is always accompanied by the formation of mainly ions containing the metal (salt cation) and acidic residue (acid anion) or its moiety [16–21]. It is most likely that the unusual scheme of the dissociative ionization of the considered complex can be explained by its electronic structure, which will be interesting to study in future.

The evaporation of $AgOOCC_6F_{11}$ (**II**) was studied in a temperature range of 367–483 K. The mass spectrum of the gas phase of $AgOOCC_6F_{11}$ is presented in Table 3. An analysis of the results given in Table 3 and the known published data on the mass spectra of the gas phase of perfluorocyclohexane, perfluorocyclohexene, and salts of perfluorocarboxylic acids [22–25] made it possible to decode the mass spectrum and conclude that the evaporation of compound **II** is accompanied by the transition to the gas phase of $CO_2$, $C_6F_{10}$, and $C_6F_{12}$ molecules and insignificant amounts of $COF_2$ and perfluorodicyclohexane ($C_6F_{11}C_6F_{11}$) molecules. The experiments on the complete isothermal sublimation of weighed samples of $AgOOCC_6F_{11}$ showed that metallic silver and trace amounts of carbon were the nonvolatile products of the evaporation of the complex determined by the X-ray fluorescence method and CHN analysis. According to the obtained

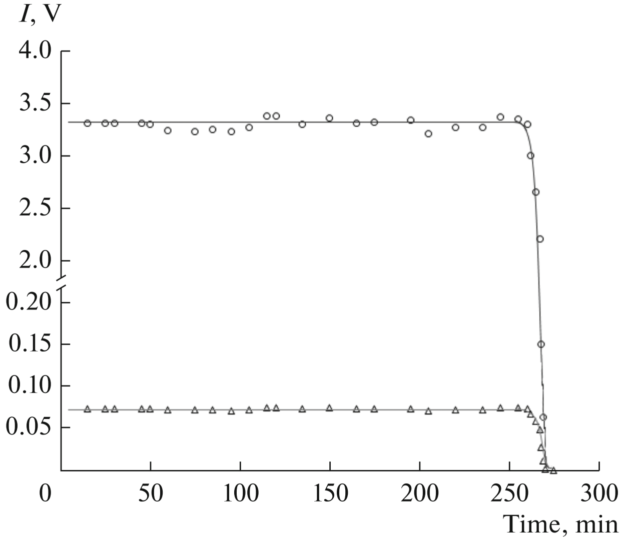

**Fig. 1.** Isotherm of the complete sublimation of the $Mo_2(PFB)_4$ weighed sample at $T = 530$ K: ○ is the ion current of $C_6F_5CO^+$, and △ is the ion current of $Mo_2(PFB)_3^+$.

**Table 2.** Enthalpies of sublimation of dimolybdenum perfluorotetrabenzoate in a temperature range of 488–555 K

| $\Delta_s H^\circ_T$, kJ/mol | | |
|---|---|---|
| $C_6F_5CO^+$ | $C_6F_5^+$ | $C_3F_4^+$ |
| 172.0 ± 4.3 | 179 ± 10 | 190.1 ± 4.5 |
| 208.1 ± 2.3 | 209.9 ± 2.7 | 211.4 ± 4.0 |
| 171 ± 20 | 160 ± 28 | 187 ± 22 |
| 218.2 ± 2.1 | 207.7 ± 3.8 | 215.4 ± 8.1 |
| 185.3 ± 4.7 | 198.4 ± 4.0 | 195.2 ± 6.6 |
| 207.3 ± 7.4 | 200 ± 10 | 205.1 ± 7.9 |
| 171.8 ± 2.3 | 180.2 ± 5.1 | 210.5 ± 6.1 |
| 191 ± 20 (average) | 191 ± 18 | 202 ± 11 |
| Recommended value | | 191 ± 20 |

data, the evaporation is mainly described by the reaction

$$2AgOOCC_6F_{11}(s) = 2Ag(s) + C_6F_{10}(g) + C_6F_{12}(g) + 2CO_2. \quad (5)$$

However, since small amounts of the $COF_2$ and $C_6F_{11}C_6F_{11}$ molecules are detected in the gas phase and the nonvolatile residue contains carbon, the following reactions can occur to an insignificant extent:

$$2AgOOCC_6F_{11}(s) = 2Ag(s) + C_6F_{11}C_6F_{11}(g) + 2CO_2, \quad (6)$$

$$AgOOCC_6F_{11}(s) = AgF(s) + C(s) + C_5F_8O(g) + COF_2(g). \quad (7)$$

The formation of acyl fluoride upon the thermal decomposition of perfluorocarboxylic acids is mentioned in many works [26, 27].

The enthalpy of reaction (5) was determined from the temperature dependence of the equilibrium constant of reaction (5) written for the corresponding ion currents ($k(5) = I_{C_6F_9}I_{C_6F_{10}}I^2_{CO_2}T^4$) using the van't Hoff equation and the least-square method in a temperature range of 367–443 K: $\Delta_r H^\circ_T(5) = 439.5 \pm 16.4$ kJ/mol (Fig. 2). Taking into account the low experimental temperatures and assuming the zero difference between the heat capacities in the first approximation, we accepted that the determined value corresponds to 298 K. The standard enthalpy of formation of complex **II** was determined from thus found enthalpy of reaction (5), the known standard enthalpies of formation of perfluorocyclohexene [28] ($\Delta_f H^\circ_{298.15}(C_6F_{10}, g) = -1906.6 \pm 7.2$ kJ/mol), perfluorocyclohexane [28] ($\Delta_f H^\circ_{298.15}(C_6F_{12}, g) = -2368.9 \pm 7.6$ kJ/mol), and carbon dioxide [22] ($\Delta_f H^\circ_{298.15}(CO_2, g) = -393.5 \pm 0.04$ kJ/mol) by the Hess law: $\Delta_f H^\circ_{298.15}(AgOOCC_6F_{11}, c) = -2751.0 \pm 24.4$ kJ/mol. Its enthalpy was calculated similarly from the temperature dependence of the equilibrium constant of reaction (6) written for the corresponding ion currents ($k(6) = I_{C_{12}F_{22}}I^2_{CO_2}T^3$): $\Delta_r H^\circ_{298.15}(6) = 325.2 \pm 14.0$ kJ/mol. Using this value and standard enthalpies of formation of $AgOOCC_6F_{11}$(c) and $CO_2$ [22], we determined the standard enthalpy of formation of bicycloperfluorohexane: $\Delta_f H^\circ_{298.15}(C_{12}F_{22}, g) = -4389.8 \pm 19$ kJ/mol, which is satisfactorily consistent with the published data: $\Delta_f H^\circ_{298.15}(C_{12}F_{22}, g) = -4363.9$ kJ/mol [29].

A relatively low determination accuracy of the standard enthalpies of formation of $AgOOCC_6F_{11}$(c) and $C_{12}F_{22}$(g) is explained by a high error in the enthalpies of reactions (5) and (6) determined by the second law of thermodynamics when studying the temperature

**Table 3.** Mass spectrum of the gas phase of $AgOOCC_6F_{11}$ ($U_{ioniz} = 70$ V)

| $m/z$ | Ion | Intensity, rel. units | $m/z$ | Ion | Intensity, rel. units |
|---|---|---|---|---|---|
| 47 | $COF^+$ | 5 | 193 | $C_5F_7^+$ | 30 |
| 88 | $C_2F_3O^+$ | 88 | 212 | $C_5F_8^+$ | 7 |
| 112 | $C_3F_4^+$ | 15 | 224 | $C_6F_8^+$ | 3 |
| 131 | $C_3F_5^+$ | 40 | 243 | $C_6F_9^+$ | 65 |
| 143 | $C_4F_5^+$ | 38 | 262 | $C_6F_{10}^+$ | 23 |
| 150 | $C_3F_6^+$ | 16 | 281 | $C_6F_{11}^+$ | 0.2 |
| 162 | $C_4F_6^+$ | 100 | 562 | $C_6F_{11}C_6F_{11}^+$ | 0.7 |

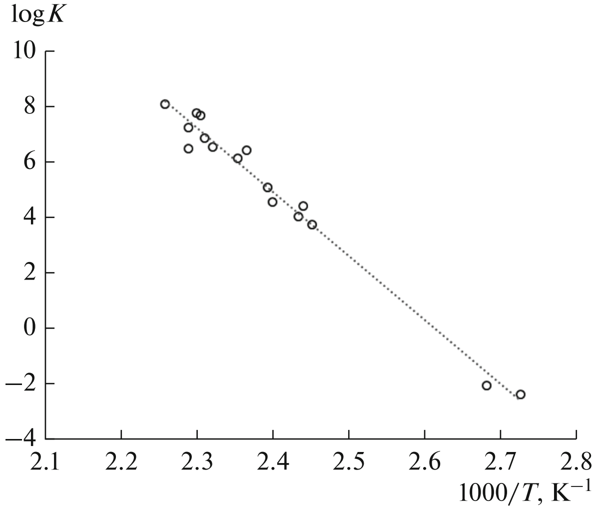

**Fig. 2.** Temperature dependence of the mass spectral equilibrium constant of reaction (5).

dependences of the gas components ($C_6F_{10}$, $C_6F_{12}$, $CO_2$), the precise measurement of which in a mass spectral experiment is very difficult. Nevertheless, the determined thermodynamic characteristics of the silver complex make it possible to recommend it for the solid-phase synthesis and determination of the thermodynamic characteristics of metal perfluorocyclohexanoates [16].

## CONCLUSIONS

(1) Anhydrous dimolybdenum perfluorotetrabenzoate $Mo_2(OOCC_6F_5)_4$ and silver perfluorocyclohexanoate $AgOOCC_6F_{11}$ were synthesized.

(2) The sublimation of the molybdenum complex was found to proceed congruently, and the saturated vapor consists of only one type of molecules: $Mo_2(OOCC_6F_5)_4$. The standard enthalpy of sublimation of the molybdenum complex and the equation of the temperature dependence of the saturated vapor pressure were found.

(3) The evaporation of silver perfluorocyclohexanoate was shown to be accompanied by the complete thermal decomposition with the formation of solid silver and mainly $C_6F_{12}$, $C_6F_{10}$, and $CO_2$ molecules. The standard enthalpy of this reaction was calculated.

(4) The standard enthalpy of formation of crystalline silver perfluorocyclohexanoate was determined.

## ACKNOWLEDGMENTS

The studies were carried out using the equipment of the Center for Collective Use of Physical Methods of Investigation at the Kurnakov Institute of General and Inorganic Chemistry (Russian Academy of Sciences).

## FUNDING

This work was supported by the Russian Science Foundation, project no. 21-13-00086.

## CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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*Translated by E. Yablonskaya*