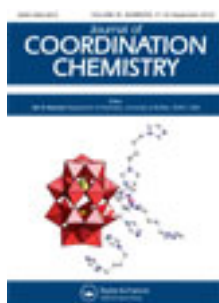


This article was downloaded by: [Renmin University of China]

On: 13 October 2013, At: 10:38

Publisher: Taylor & Francis

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Journal of Coordination Chemistry

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/gcoo20>

Syntheses, structures, and thermal properties of new Au(III) organometallic compounds with ancillary S-, O-, and/or N-donor ligands

R. Parkhomenko^a, A. Alexeyev^a, N. Morozova^a & I. Igumenov^a

^a Nikolaev Institute of Inorganic Chemistry, Siberian Branch of Russian Academy of Sciences, Lavrentiev avenue, 3, Novosibirsk 630090, Russia

Accepted author version posted online: 18 Jul 2012. Published online: 01 Aug 2012.

To cite this article: R. Parkhomenko, A. Alexeyev, N. Morozova & I. Igumenov (2012) Syntheses, structures, and thermal properties of new Au(III) organometallic compounds with ancillary S-, O-, and/or N-donor ligands, Journal of Coordination Chemistry, 65:18, 3227-3237, DOI: [10.1080/00958972.2012.713476](https://doi.org/10.1080/00958972.2012.713476)

To link to this article: <http://dx.doi.org/10.1080/00958972.2012.713476>

PLEASE SCROLL DOWN FOR ARTICLE

Taylor & Francis makes every effort to ensure the accuracy of all the information (the "Content") contained in the publications on our platform. However, Taylor & Francis, our agents, and our licensors make no representations or warranties whatsoever as to the accuracy, completeness, or suitability for any purpose of the Content. Any opinions and views expressed in this publication are the opinions and views of the authors, and are not the views of or endorsed by Taylor & Francis. The accuracy of the Content should not be relied upon and should be independently verified with primary sources of information. Taylor and Francis shall not be liable for any losses, actions, claims, proceedings, demands, costs, expenses, damages, and other liabilities whatsoever or howsoever caused arising directly or indirectly in connection with, in relation to or arising out of the use of the Content.

This article may be used for research, teaching, and private study purposes. Any substantial or systematic reproduction, redistribution, reselling, loan, sub-licensing, systematic supply, or distribution in any form to anyone is expressly forbidden. Terms &

Conditions of access and use can be found at <http://www.tandfonline.com/page/terms-and-conditions>

Syntheses, structures, and thermal properties of new Au(III) organometallic compounds with ancillary S-, O-, and/or N-donor ligands

R. PARKHOMENKO*, A. ALEXEYEV, N. MOROZOVA and I. IGUMENOV

Nikolaev Institute of Inorganic Chemistry, Siberian Branch of Russian Academy of Sciences, Lavrentiev avenue, 3, Novosibirsk 630090, Russia

(Received 16 May 2012; in final form 20 June 2012)

$((\text{CH}_3)_2\text{Au})_2\text{C}_2\text{O}_4$ (**1**), $((\text{CH}_3)_2\text{AuSCN})_2$ (**2**), $(\text{CH}_3)_2\text{AuSSP}(\text{OCH}_3)_2$ (**3**), and $(\text{CH}_3)_2\text{AuSSP}(\text{OC}_2\text{H}_5)_2$ (**4**) were prepared and recrystallized from hexane to determine their crystal structures and analyze them by thermal methods (TGA). The compounds have been investigated as new possible precursors for metal–organic chemical vapor deposition (MOCVD). Compounds **1** and **2** are solids, while **3** and **4** are liquids. Crystal structures of **1** and **2** have been studied by single-crystal X-ray diffraction (XRD): compounds are monoclinic, space group for **1** $P2_1/c$, for **2** $P2_1/n$. Compound **1** has crystal parameters $a = 7.6952(5) \text{ \AA}$, $b = 11.1814(8) \text{ \AA}$, $c = 12.2893(8) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 104.922(4)^\circ$, $\gamma = 90^\circ$; **2** has crystal parameters $a = 5.6184(3) \text{ \AA}$, $b = 15.2744(6) \text{ \AA}$, $c = 6.9202(3) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 102.864(2)^\circ$, $\gamma = 90^\circ$. These are neutral complexes, in which molecules are only connected by van der Waal's interactions. Thermal gravimetric analyses (TGA) have shown that **3** and **4** evaporate practically without decomposition. MOCVD experiments were carried out at lower pressure using **3** and **4** as precursors. The films were grown on Si substrate and investigated by XRD and SEM.

Keywords: Gold compound; MOCVD; Thin films

1. Introduction

Thin gold films are used in electronics as a contact material, as a material for corrosion-resistant coatings within microwave resonators, and as internal surfaces of waveguides [1, 2]. A considerable advantage of gold as the contact material consists in its resistance against formation of sulfurous and oxide films under atmospheric conditions either at room temperature or on heating. Gold nanoparticles exhibit a high catalytic activity in chemical processes, such as the oxidation of hydrocarbons, the oxidation of methanol to give formaldehyde, and low-temperature CO oxidation [3–5]. Nanostructured materials based on gold have become the object of extensive studies in contemporary medicine [6–9].

One way to obtain thin gold films and nanoparticles is by metal–organic chemical vapor deposition (MOCVD). Volatile organometallic Au(III) compounds are used as

*Corresponding author. Email: parkhomenko@niic.nsc.ru

precursors [10–14], and those must meet certain requirements, such as a high vapor pressure at low temperature, and stability in the condensed and gaseous phases [15]. The content of gold in molecules of known precursors ranges from 50% to 69% (mass percentage) [10–14]. Searching for compounds with a higher content of gold represents an urgent problem. Moreover, complexes used in MOCVD systems under standard conditions represent solid substances and thus there are certain difficulties with dosing them. The use of liquid precursor compounds allows a much more accurate dosing of the compound vapor concentration within the reaction zone. In addition, the use of liquid precursors allows one to obtain gold films using the method of aerosol spraying without solvents employed [16].

This work consisted in synthesizing of Au(III) compounds, identifying them, obtaining crystal chemical data, studying the thermal behavior in the condensed phase (thermal gravimetric analyses, TGA), performing MOCVD test experiments, and characterization of coatings obtained.

2. Experimental

2.1. Materials

The chemicals benzene (Chemreactive, Russia, purity >99%), hexane (Chemreactive, Russia, purity >99%), diethyl ether (Chemreactive, Russia, purity >99%), THF (Chemreactive, Russia, purity >98%), KSCN (Chemreactive, Russia, purity >98%), $\text{Na}_2\text{C}_2\text{O}_4$ (Chemreactive, Russia, purity >97%), AgNO_3 (Chemreactive, Russia, purity >98%), $\text{KSSP}(\text{OC}_2\text{H}_5)_2$ (Merck, purity >98%), and $\text{KSSP}(\text{OC}_2\text{H}_5)_2$ (Merck, purity >98%) were used in syntheses of gold complexes. All solvents were purified and dried. Elemental analyses were performed on a EuroEA3000, Eurovector. IR spectra were recorded from 400 to 4000 cm^{-1} on a Scimitar FTS2000 spectrometer using KBr pellets or liquid film between KBr plates.

2.2. Synthesis of complexes

2.2.1. Synthesis of $(\text{CH}_3)_2\text{AuI}$. A mixture of 0.92 g of magnesium powder (0.038 mol) and 10 mL of dry diethyl ether was added into a flat-bottomed cone flask with a backflow condenser and a dropping funnel. The mixture was heated to 40°C and then gradually to this, within 1 h, a solution of 5.45 g (0.038 mol) of methyl iodide in 40 mL of dry diethyl ether was added dropwise, under stirring. After the reaction was completed, 50 mL of benzene was added and the mixture was cooled to -30°C . Dried KAuCl_4 (3.63 g, 0.01 mol) was added to the cooled mixture under intense stirring. Within 30 min after adding KAuCl_4 , the reaction mixture was slowly warmed to -10°C for further decomposing by HCl-acidified water (pH ~ 2) with ice. An organic portion was separated and an aqueous part was extracted with hexane ($3 \times 150\text{ mL}$). The organic portion was evaporated under atmospheric pressure and the product was purified using recrystallization from hexane. The yield was 53% (1.8 g).

2.2.2. Synthesis of $((\text{CH}_3)_2\text{Au})_2\text{C}_2\text{O}_4$ (1). To a solution $(\text{CH}_3)_2\text{AuI}$ (0.2 g) in 30 mL of benzene was added silver oxalate (0.1 g) at a ratio of 1:1 between reagents, under intense stirring. The reaction was performed for 1 h at room temperature. After the reaction was completed, the solution was filtered, then solvent was removed under reduced pressure. The precipitate obtained was recrystallized from hexane. The complex was transparent needle-shaped crystals with a melting point of 77°C. The compound is soluble in common organic solvents. The yield of the complex was 85%. IR (cm^{-1}): 2923(s), 2854(m), 1623(m), 1593(m), 1461(m), 1410(m), 1213(m), 1184(m), 794(m), 450(m). Anal. Calcd for $\text{C}_6\text{H}_{12}\text{Au}_2\text{O}_4$ (%): C, 13.3; H, 2.21. Found: C, 12.9; H, 2.05.

2.2.3. Synthesis of $((\text{CH}_3)_2\text{AuSCN})_2$ (2). To a solution of $(\text{CH}_3)_2\text{AuI}$ (0.2 g) in 30 mL of benzene was added thoroughly dried silver thiocyanate (0.46 g) at a ratio of 1:5 between reagents, under intense stirring. The reaction was carried out for 2.5 h at room temperature. After the reaction was completed, the solution was filtered, and solvent removed under atmospheric pressure. The precipitate was recrystallized from hexane. The complex was white needle-shaped crystals with melting point of 80°C. The compound is highly soluble in common organic solvents. The yield was 75%. IR (cm^{-1}): 2922(s), 2854(m), 2163(s), 1462(w), 1377(w), 1240(m), 1198(m), 808(w), 774(w), 442(w). Anal. Calcd for $\text{C}_6\text{H}_{12}\text{Au}_2\text{S}_2\text{N}_2$ (%): C, 12.6; H, 2.11; N, 4.91; S, 11.23. Found: C, 12.4; H, 2.05; N, 4.85; S, 11.06.

2.2.4. Synthesis of $(\text{CH}_3)_2\text{AuSSP}(\text{OCH}_3)_2$ (3). To a solution of Me_2AuI (0.2 g) in 10 mL of THF was added a solution of $\text{KSSP}(\text{OCH}_3)_2$ (0.12 g) in 20 mL of THF at a ratio of 1:1. The reaction mixture was stirred at room temperature for 1 h. After the reaction was completed, the solvent was removed under atmospheric pressure and product was purified using column chromatography with SiO_2 sorbent. Under standard conditions, the complex is a transparent oily liquid. The yield of the complex was 85%. IR (cm^{-1}): 2920(s), 2852(s), 1457(m), 1222(m), 1189(m), 1023(vs), 818(s), 802(s), 655(m), 639(m), 524(w). Anal. Calcd for $\text{C}_4\text{H}_{12}\text{AuPS}_2\text{O}_2$ (%): C, 12.5; H, 3.13. Found: C, 12.4; H, 3.09.

2.2.5. Synthesis of $(\text{CH}_3)_2\text{AuSSP}(\text{OC}_2\text{H}_5)_2$ (4). To a solution of $(\text{CH}_3)_2\text{AuI}$ (0.2 g) in 10 mL of THF was added a solution of $\text{KSSP}(\text{OC}_2\text{H}_5)_2$ (0.12 g) in 20 mL of THF at a ratio of 1:1. The reaction mixture was stirred at room temperature for 1 h. After the reaction was completed, the solvent was removed under atmospheric pressure and the product purified using column chromatography with SiO_2 sorbent. Under standard conditions, the complex is a transparent oily liquid. The yield of the complex was 83%. IR (cm^{-1}): 2985(s), 2914(s), 2855(m), 1473(m), 1442(m), 1390(m), 1222(m), 1191(m), 1162(m), 1014(vs), 968(vs), 815(s), 792(s), 649(s), 636(s), 543(m). Anal. Calcd for $\text{C}_6\text{H}_{16}\text{AuPS}_2\text{O}_2$ (%): C, 17.5; H, 3.88. Found: C, 17.3; H, 3.81.

2.3. Thermal analysis

TGA data were obtained using a Netzsch TG 209 F1 Iris[®] device. Experiments were conducted in He flow (70 mL min^{-1}) with heating rate 1 K min^{-1} from 25 to 300°C. Samples of 5.0–10.0 mg were loaded in α -alumina crucibles ($5 \times 5\text{ mm}$).

2.4. X-ray diffraction analyses

The X-ray diffraction (XRD) analyses of the samples were performed at room temperature on a DRON-3M diffractometer (Cu-K α radiation, graphite monochromator on the reflected beam, and a scintillation detector with amplitude discrimination). The spectra were recorded in the step-by-step mode in the angular range $2\theta = 25\text{--}65^\circ$.

2.5. Scanning electron microscopy images

Surfaces of gold samples were investigated by scanning electron microscopy (SEM) with SEM images of films recorded with a JEOL-JSM 6700F.

2.6. X-ray crystallographic study

The structures of **1** and **2** were solved by single-crystal XRD analyses. Suitable single crystals were mounted on a Bruker Nonius X8 Apex 4K CCD diffractometer fitted with graphite monochromated Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$). The data were collected at 150 K for all structures by the standard technique [17]. Absorption corrections were made empirically using SADABS [17]. Structures were solved by the direct methods of the difference Fourier synthesis and further refined by the full-matrix least-squares using SHELXTL [18]. Positions of hydrogen atoms were calculated geometrically and refined in the rigid body approximation (riding model). The crystallographic data, selected bond lengths, and angles for **1** and **2** are given in tables 1 and 2.

2.7. Deposition procedure

Compounds **3** and **4** were used as precursors for deposition of gold films. MOCVD experiments were carried out in LPCVD at 10 Torr using Ar as a carrier gas (flow 25 mL min^{-1}). The films were deposited on Si substrates. The evaporator temperature for **3** was 55°C and for **4** was 60°C . The substrate temperature for both compounds varied from 210°C to 250°C . All deposition experiments were carried out for 1 h.

3. Results and discussion

3.1. Synthesis

The complexes were synthesized *via* the exchange reaction between $(\text{CH}_3)_2\text{AuI}$ and silver/potassium salt of a corresponding ligand and are shown in the following scheme.



$\text{Ag}_2\text{C}_2\text{O}_4$ was obtained *via* reaction between $\text{Na}_2\text{C}_2\text{O}_4$ and AgNO_3 in aqueous solution. AgSCN was obtained *via* reaction between KSCN and AgNO_3 in aqueous solution.

Table 1. Crystal data and structure refinements for $((\text{CH}_3)_2\text{Au})_2\text{C}_2\text{O}_4$ (**1**) and $((\text{CH}_3)_2\text{AuSCN})_2$ (**2**).

Formula	$\text{C}_3\text{H}_6\text{AuO}_2$	$\text{C}_3\text{H}_6\text{AuNS}$
Formula weight	271.04	285.11
Temperature (K)	150(2)	150(2)
Wavelength (\AA)	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/c$	$P2_1/n$
Unit cell dimensions (\AA , $^\circ$)		
<i>a</i>	7.6952(5)	5.6184(3)
<i>b</i>	11.1814(8)	15.2744(6)
<i>c</i>	12.2893(8)	6.9202(3)
α	90	90
β	104.922(4)	102.864(2)
γ	90	90
Volume (\AA^3), <i>Z</i>	1021.75(12), 8	578.97(5), 4
Calculated density (Mg m^{-3})		
Absorption coefficient (mm^{-1})	28.659	25.625
<i>F</i> (000)	952	504
Crystal size (mm^3)	$0.25 \times 0.03 \times 0.03$	$0.20 \times 0.02 \times 0.02$
θ range for data collection	2.50–26.37	2.67–26.37
Limiting indices	$-9 \leq h \leq 9$; $-13 \leq k \leq 13$; $-13 \leq l \leq 15$	$-7 \leq h \leq 7$; $-19 \leq k \leq 19$; $-8 \leq l \leq 8$
Reflections collected/unique (<i>R</i> _{int})	2082 [<i>R</i> (int) = 0.0827]	1182 [<i>R</i> (int) = 0.0589]
Completeness to $\theta = 25.00^\circ$ (%)	99.9	100.0
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.4802 and 0.0526	0.6282 and 0.0796
Refinement method	Full-matrix least squares on <i>F</i> ²	
Data/restraints/parameters	2082/0/114	1182/0/58
Goodness-of-fit on <i>F</i> ²	1.012	1.249
Final <i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> ₁ = 0.0435, <i>wR</i> ₂ = 0.0842	<i>R</i> ₁ = 0.0295, <i>wR</i> ₂ = 0.0476
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0669, <i>wR</i> ₂ = 0.0893	<i>R</i> ₁ = 0.0316, <i>wR</i> ₂ = 0.0483
Largest difference in peak and hole (e \AA^{-3})	2.118 and -2.044	0.773 and -1.187

Table 2. Selected bond distances (\AA) and angles ($^\circ$).

$((\text{CH}_3)_2\text{Au})_2\text{C}_2\text{O}_4$ (1)		$((\text{CH}_3)_2\text{AuSCN})_2$ (2)	
Au(1)–C(12)	2.013(15)	Au(1)–C(1)	2.041(7)
Au(1)–C(11)	2.031(13)	Au(1)–C(2)	2.064(7)
Au(1)–O(11)	2.143(8)	Au(1)–N(1)	2.104(7)
Au(1)–O(12)	2.153(8)	Au(1)–S(1)	2.455(2)
Au(2)–C(22)	2.020(14)	N(1)–C(3)	1.154(9)
Au(2)–C(21)	2.030(13)		
Au(2)–O(22)	2.151(9)		
Au(2)–O(21)	2.161(9)		
O(11)–C(13)	1.269(15)		
O(22)–C(23)	1.240(15)		
C(12)–Au(1)–C(11)	87.4(6)	C(1)–Au(1)–C(2)	86.3(3)
C(12)–Au(1)–O(11)	96.7(5)	C(2)–Au(1)–N(1)	92.4(3)
C(11)–Au(1)–O(12)	97.2(5)	C(1)–Au(1)–S(1)	88.8(2)
O(11)–Au(1)–O(12)	78.8(3)	N(1)–Au(1)–S(1)	92.51(17)
C(13)–O(11)–Au(1)	112.7(8)	C(3)–N(1)–Au(1)	166.0(6)

3.2. Descriptions of the structures

3.2.1. $((\text{CH}_3)_2\text{Au})_2\text{C}_2\text{O}_4$ (1**).** $((\text{CH}_3)_2\text{Au})_2\text{C}_2\text{O}_4$ is crystallized as transparent colorless crystals with acicular (needle-like) habitus. The crystal structure consists of neutral molecules. Gold exhibits a slightly distorted square-planar coordination formed by two carbon atoms of methyl groups and two oxygen atoms from oxalate (figure 1a). In this structure there are two crystallographically independent molecules, exhibiting different orientations in the unit cell. The Au–O distances vary from 2.143 to 2.161 Å, average to 2.152 Å, which is typical for dimethylgold(III) carboxylates. In this case, chelate angles O–Au–O appear equal to be less than 90° (78.8° and 78.6° for the first and the second crystallographically independent molecules, respectively).

The average Au–CH₃ bond length is 2.023 Å, the C–Au–C angle also differs from 90° by 3.1° , the same value was observed in the literature for other dimethylgold(III) systems [14, 19–22]. The bending angle between planes C11–Au1–C12 and O11–Au1–O12 in the first molecule, and between planes C21–Au2–C22 and O21–Au2–O22 in the second, differ to a considerable extent, 1.277° and 0.843° , respectively. This structure exhibits a parquet type (figure 1b) packing of molecules, as against other known dimethylgold(III) carboxylates, wherein the molecules are packed to form infinite 1-D chains [19, 21]. The molecules within one pseudo-layer are associated due to van der Waals interactions. The distance between Au1 and Au2 in neighboring molecules of one pseudo-layer is 3.470 Å, van der Waals radius for Au ~ 1.7 .

3.2.2. $((\text{CH}_3)_2\text{AuSCN})_2$ (2**).** $((\text{CH}_3)_2\text{AuSCN})_2$ crystallizes in the form of transparent colorless crystals with acicular (needle-like) habitus with the structure consisting of neutral molecules. Gold exhibits a slightly distorted square-planar coordination formed by two carbon atoms of methyl groups and nitrogen and sulfur of thiocyanate (figure 2a). The Au1–S1 distance is 2.455 Å, which is 0.351 Å longer than the Au1–N1 distance, similar to other S, N-coordinated dimethylgold(III) compounds [20]. The average value for Au–CH₃ bond lengths is 2.053 Å, somewhat longer than $((\text{CH}_3)_2\text{Au})_2\text{C}_2\text{O}_4$ and longer than the values of 2.02–2.04 Å well-known from the literature [19–22]. The reason for this is the *trans* influence of the terminal sulfur, which is much stronger than terminal oxygen [23]. The C1–Au–C2 angles are equal to 86.3° , whereas the N1–Au–S1 angle is 92.51° . This crystal structure exhibits a parquet type of packing (figure 2b). Molecules within one pseudo-layer are bound *via* van der Waals interactions. The distance between gold Au \cdots Au within one pseudo-layer is 3.839 Å.

3.3. Thermogravimetric study of synthesized complexes

Curves of mass loss in the course of heating the complexes in an atmosphere of helium are provided in “Supplementary material.” Under the experimental conditions, **1** began to decompose at 75°C in two steps; the mass loss in the first step is 15% and 14% at the second stage. The mass content of gold in the molecule is 73%, while mass loss of **1** at the final temperature is 44%, indicating that there is transfer of the compound to the gas phase.

Decomposition of **2** occurs at 106°C with 24.5% mass loss, whereas the mass content of gold is 69%. The increase in mass could be connected with incomplete decomposition

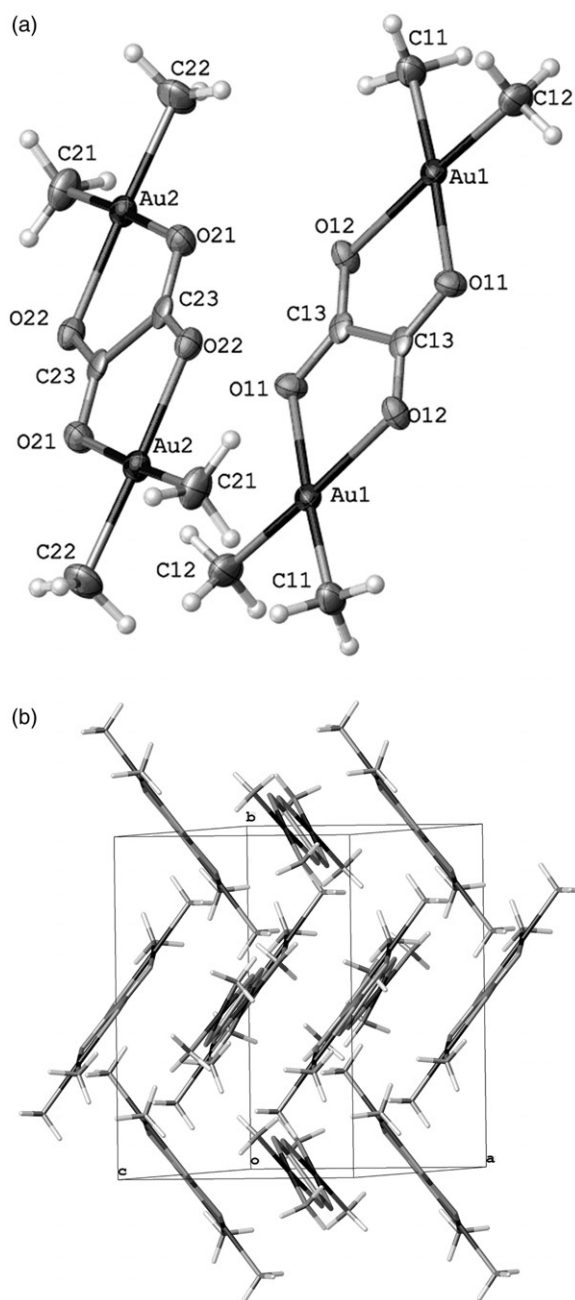
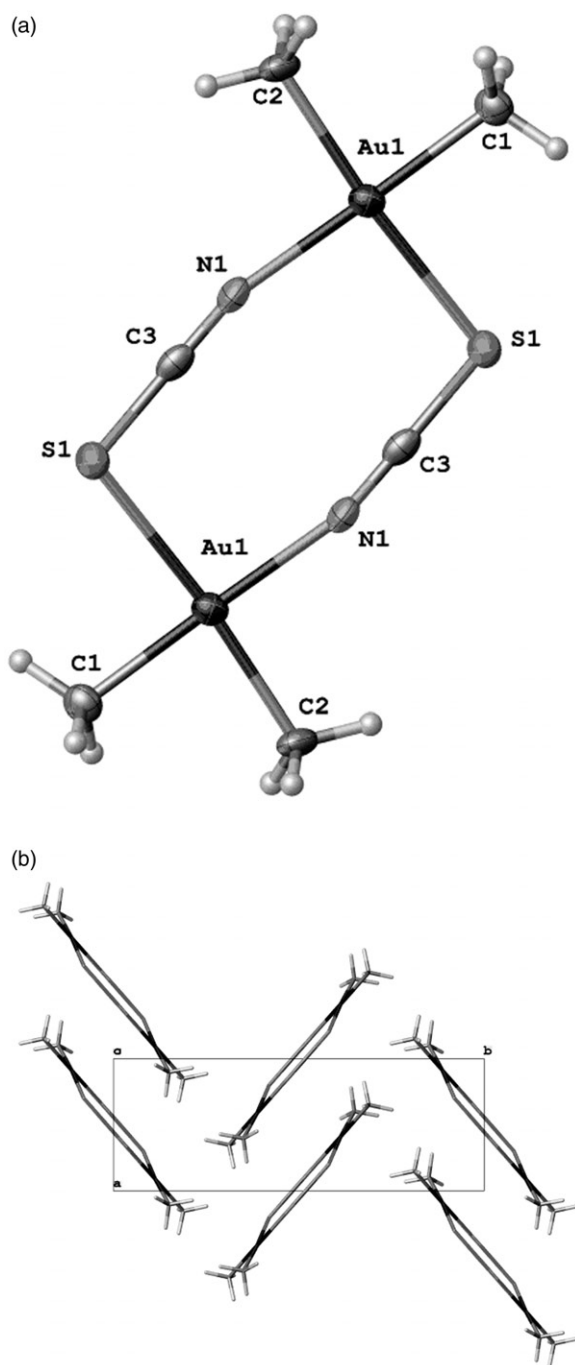


Figure 1. Structure (a) and packing (b) of $((\text{CH}_3)_2\text{Au})_2\text{C}_2\text{O}_4$ (1).

of organic fragments. According to the XRD phase analysis, the main component of the residue is metallic gold.

The mass loss of **3** is 87%. Compound **4** is transferred to the gas phase under the thermogravimetric conditions almost without decomposition with mass loss being 93%.



Based on the thermal studies we have chosen **3** and **4** as precursors for the MOCVD test experiments.

3.4. MOCVD experiments

MOCVD test experiments were performed employing an LPCVD flow-through reactor using volatile **3** and **4** as precursors. Based on the TG curves, the temperature of evaporator was chosen to be 55°C for **3** and 60°C for **4**. According to the TG data the mass loss value for **3** was 87%, whereas in MOCVD, the mass loss is greater than 95%. The discrepancy could be explained by a high heating rate of the compound in the thermogravimetric method. When the temperature reaches 110°C, the material begins to decompose with incomplete evaporation, indicated by abrupt changing in the slope of the mass loss curve.

Figure 3 demonstrates SEM images for two samples deposited at 250°C. According to SEM, the average crystallite size for the films deposited from **3** is 150 nm, whereas that for **4** is 50 nm at the mentioned deposition temperature.

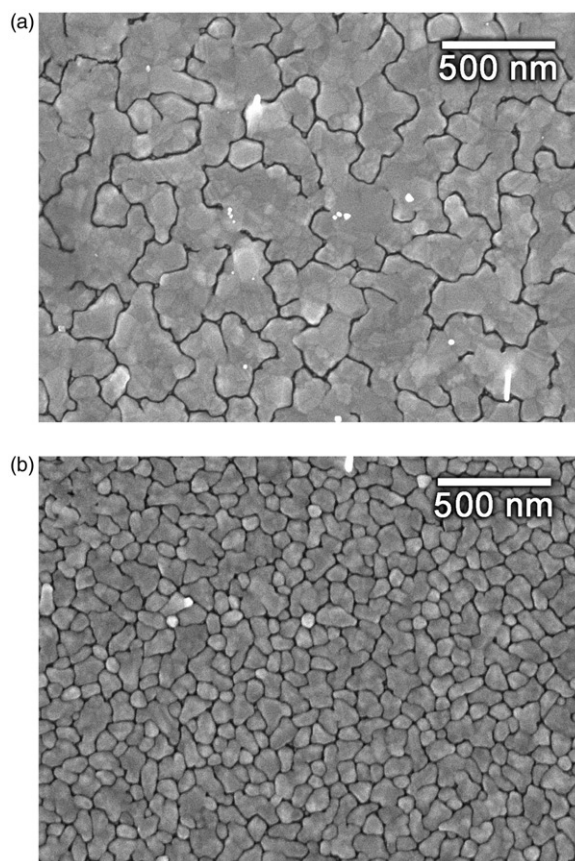


Figure 3. SEM photographs of thin gold films deposited from **3** (a), and from **4** (b) at 250°C.

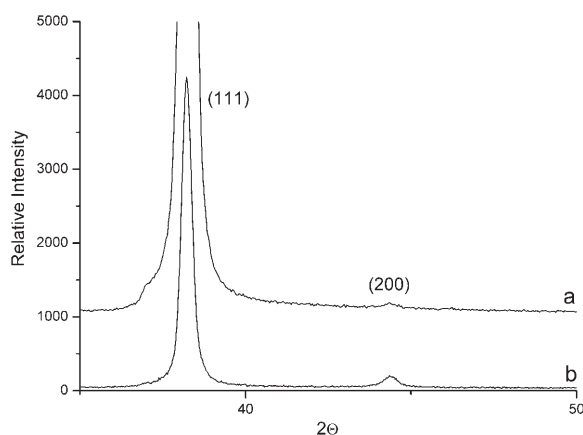


Figure 4. XRD data for thin gold films deposited from **3** (a), and from **4** (b) at 250°C.

According to the XRD phase analysis, the growth of crystallites in the samples under investigation occurs with preferred orientation in the crystallographic direction [111] (figure 4). Parameters of the unit cell of gold in the samples did not differ from the tabulated value of 4.0786 Å [24]. The $I_{\text{Au}(200)}/I_{\text{Au}(111)}$ intensity ratio for sample (a) is 0.00169, whereas that for sample (b) is 0.03694.

4. Conclusion

Four complexes of dimethylgold(III) with ancillary S-, O-, and/or N-donors, $((\text{CH}_3)_2\text{Au})_2\text{C}_2\text{O}_4$ (**1**), $((\text{CH}_3)_2\text{AuSCN})_2$ (**2**), $(\text{CH}_3)_2\text{AuSSP}(\text{OCH}_3)_2$ (**3**), and $(\text{CH}_3)_2\text{AuSSP}(\text{OC}_2\text{H}_5)_2$ (**4**), have been synthesized and characterized. Complexes **1** and **2** are solids, whereas **3** and **4** are liquids under standard conditions. XRD structural investigations were performed for **1** and **2**. The thermal stability of the synthesized compounds in the condensed phase was studied using thermogravimetry; **3** and **4** transfer to the gas phase with almost no decomposition, whereas **1** and **2** decompose on heating. For volatile liquids **3** and **4** MOCVD test experiments were performed. According to SEM, the average crystallite size for the films deposited from $(\text{CH}_3)_2\text{AuSSP}(\text{OCH}_3)_2$ (**3**) is 150 nm and from $(\text{CH}_3)_2\text{AuSSP}(\text{OC}_2\text{H}_5)_2$ is 50 nm at 250°C. According to the XRD analysis, the deposited gold samples demonstrate crystallite growth mainly in the [111] direction.

Supplementary material

CCDC no. 881 760 for **1** and no. 881 761 for **2** contain the supplementary crystallographic data. These data can be obtained *via* the Cambridge Crystallographic Data Centre (deposit@ccdc.cam.ac.uk; <http://www.ccdc.cam.ac.uk/deposit>).

References

- [1] A. Sherman. *Chemical Vapor Deposition for Microelectronics*, Noyes Publications, New Jersey (1987).
- [2] M. Oren, A.N.M. Masum Choudhury. *J. Electrochem. Soc.*, **134**, 750 (1987).
- [3] H. Haruta, S. Tsubota, T. Kobayashi, H. Kageyama, M.J. Genet, B. Delmon. *J. Catal.*, **144**, 175 (1993).
- [4] M. Okumura, K. Tanaka, A. Ueda, M. Haruta. *Solid State Ionics*, **95**, 143 (1997).
- [5] M. Haruta. *Catal. Today*, **36**, 153 (1997).
- [6] U. Kreibig, M. Vollmer. *Optical Properties of Metal Clusters*, Springer-Verlag, Berlin (1995).
- [7] H. Wang, D.W. Brandl, F. Le, P. Nordlander, N.J. Halas. *Nano Lett.*, **6**, 827 (2006).
- [8] N.G. Khlebtsov, A.G. Mel'nikov, V.A. Bogatyrev, L.A. Dykman, A.V. Alekseeva, L.A. Trachuk, B.N. Khlebtsov. *J. Phys. Chem. B*, **109**, 13578 (2005).
- [9] G. Ramakrishna, Q. Dai, J. Zou, Q. Huo, T.J. Goodson. *J. Am. Chem. Soc.*, **129**, 1848 (2007).
- [10] A.A. Bessonov, N.B. Morozova, N.V. Gelfond, P.P. Semyannikov, S.V. Trubin, Yu.V. Shevtsov, Yu.V. Shubin, I.K. Igumenov. *Surf. Coat. Technol.*, **201**, 9099 (2007).
- [11] A. Turgambaeva, G. Zharkova, P. Semyannikov, V.V. Krisyuk, T. Koretskaya, S. Trubin, B. Kuchumov, I. Igumenov. *Gold Bull.*, **4**, 177 (2011).
- [12] C.E. Larson, T.H. Baum, R.L. Jackson. *J. Electrochem. Soc.*, **134**, 266 (1987).
- [13] K. Holloway, S.P. Zuhoski, S. Reynolds, C. Matuszewski. *Mater. Res. Soc. Symp. Proc.*, **204**, 409 (1991).
- [14] A.A. Bessonov, N.B. Morozova, N.V. Gelfond, P.P. Semyannikov, I.A. Baidina, S.V. Trubin, Y.V. Shevtsov, I.K. Igumenov. *J. Organomet. Chem.*, **693**, 2572 (2008).
- [15] T. Kodas, M. Hampden-Smith (Eds). *The Chemistry of Metal CVD*, Weinheim, New York, Basel, Cambridge, Tokyo (1994).
- [16] K. Fröhlich, D. Machajdík, A. Rosová, I. Vávra, F. Weiss, B. Bochu, J.P. Senateur. *Thin Solid Films*, **260**, 187 (1995).
- [17] Bruker AXS Inc. *APEX2 (Version 1.08)*, *SAINT (Version 7.03)*, and *SADABS (Version 2.11)*, Bruker Advanced X-ray Solutions, Madison, WI, USA (2004).
- [18] G.M. Sheldrick. *Acta Crystallogr.*, **A64**, 112 (2008).
- [19] A.A. Bessonov, I.A. Baidina, N.B. Morozova, P.P. Semyannikov, S.V. Trubin, N.V. Gelfond, I.K. Igumenov. *J. Struct. Chem.*, **48**, 282 (2007).
- [20] A.A. Bessonov, I.A. Baidina, N.B. Morozova, N.V. Kuratieva, N.V. Gelfond, I.K. Igumenov. *J. Struct. Chem.*, **49**, 64 (2008).
- [21] A.A. Bessonov, N.B. Morozova, N.V. Kurat'eva, I.A. Baidina, N.V. Gel'fond, I.K. Igumenov. *Russ. J. Coord. Chem.*, **34**, 70 (2008).
- [22] A.A. Bessonov, A.I. Smolentsev, I.A. Baidina, N.B. Morozova. *Acta Crystallogr., Sect. E*, **63**, 3154 (2007).
- [23] J.D. Atwood. *Inorganic and Organometallic Reaction Mechanisms*, 2nd Edn, VCH, Weinheim (1997).
- [24] Powder Diffraction File No. 4-784. *Alphabetical Index: Inorganic Phases*, JCPDS, International Centre for Diffraction Data, Pennsylvania, USA (1983).