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Complexes of Silver (I) Perchlorate and Mercury (II) Halides with Some Tertiary Arsines

KEYWORDS
silver (I) perchlorate; mercury (II) chloride and bromide; tertiary arsine; infrared spectrum; thermogravigram

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ABSTRACT *The interaction of silver (I) perchlorate and mercury (II) halides with triaryl- and diarylalkyl arsines was studied. As a result the complex compounds were obtained having the following formulas*

$[Ag((C_6H_5)_2R)_4]ClO_4$ and $HgHlg_2\,As(R)Ar_2$, were $Ar = -C_6H_5$, $m-CH_3C_6H_4$;

$R = -C_6H_5$, $m-CH_3-C_6H_4$, C_2H_5, $izo-C_3H_7$, $n-C_3H_7$, $izo-C_4H_9$.

and ,were ; , , , , , . Both the elemental and physicochemical analysis were applied to study composition and structure of the received compounds.Coordination formulas were established by measuring molar conductivity of the synthesized compounds solved in DMF (dimethylformamide). The molar conductivity of silver (I) tetra (triaryl- and diarylalkyl) arsine perchlorates ranges within 62-71 Om–1·cm2·mol–1 which corresponds to molar conductivity of double-ion type electrolytes. As for complexes of mercury (II) chloride and bromide with tertiary arsines, their molar conductivity does not exceed 30 Om–1·cm2·mol–1 , which proves the compounds being the nonelectrolytes. Study of the infrared spectrum as well as thermal and elemental analysis of the synthesized complex compounds were conducted.

Introduction.

One of the priorities in inorganic chemistry is the chemistry of complex compounds, which keeps developing quite intensive. This is determined by the almost inexhaustible ability to synthesize compounds of desired properties. New substances are synthesized by means of purposeful changes in composition and structure of initial products. This refers not only to the central complexing ion but to ligands as well, transformation of which causes the change in crystalline structure and charachteristics. From this point of view the attention should be paid to tertiary arsines and their derivatives (chalkogenides, arsenium salts, etc.) as for ligands required for synthesis of complex compounds [1]. Their chemical structure and ability to interact with other substances are close to those of ammonia; in addition, they are characterized by strongly pronounced physiological activity [2].

The ability of triaryl (alkyl) arsines to interact with other substances mostly conditioned by the existence of lone pair of electrons on the $4S^2$ subshell of the arsenic atom. While interacting with electrophilic reagents (namely haloalkanes) tertiary arsines undergo quaternization producing arsenium salt [3]. In this case the formation of the 4th bound can be considered as coordination of the electrons pair of arsenic (III) with Lewis' nucleophylic center, as a result of which the central atom obtains formally positive charge [4]. The same process can be observed during interaction of triaryl (alkyl) arsines with salts, where they represent ligands. Thereby, due to the existence of the non-bonding pair of electrons the central atom of tertiary arsines tends to form complex compounds with metal halides (especially with the salts of transition elements) [5, 6]. Various scientists made their opinion as for complex compounds being used to identify ligands and for their indirect complexometric analysis.

Experimental Procedure

For determination of percentage of arsenic in the samples the well-known Evins' method was applied [12]. Concetration of silver (I) was determined by using rhodanide method [13]; percentage of halogens – using mercurometry [14].

Spectrophotometer UR-20 was applied for receiving IR spectra of organoarsenic ligands and synthesized coordination compounds; samples were pressed in vaseline.

Thermogravimetric analysis (TGA) was performed on a Paulik-Paulik-Erday derivatograph. That enabled to receive four thermal curves at the same time, which reflected processes related to the heating of samples. Recording of the derivatograms continued for two hours at constant atmospheric pressure and humidity. DTA sensitivity of the recording was1/5, DTG sensitivity – 1/20, temperature accuracy range ±5°C. Heated aluminum oxide was used as an etalon for comparison. 100 mg of the substance was placed on a plate-form platinum holder. Applied heating rate was 10 ^{0}C min^{-1}.

Specific conductivity of the samples was determined in the refined and purified solution of dimethylformamide at the temperature 25°C. The data were taken on "Impulse" type conductometer with the measurement accuracy of ±1.5%. Molar conductivity was calculated according to the following formula:

$$\mu = \frac{100}{c} \ \text{Om}^{-1}\cdot\text{cm}^{2}\cdot\text{mol}^{-1}$$

where μ and *C* are consequently stand for specific conductivity and concentration of the solution. Constancy of temperature was secured by the application of water bath with the temperature accuracy of ±0.5°C. Specific conductivity was measured several times for each solution so that to get similar results.

Roentgenographic study of the samples were performed using diffractometer DPOH-3M in the Laboratory of Physical and Chemical Research of the Institution of Inorganic Chemistry and Electrochemistry applying CuKa radiation (30 kV, 30 mA).

All synthesized substances are characterized by crystalline structure.

Synthesis of silver (I) tetra (triphenylarsine) perchlorate $[Ag(As(C_6H_5)_3)_4]ClO_4$. 4 g. (0.0131 mol) of triphenylarsine is solved in 50 ml. of tetrahydrofuran. To the received solution, while constantly stirring, 0.68 g. (0.0033 mol) of saturated etheric solution of silver (I) perchlorate is added. The residue of the white colour is obtained, which is then settled for a night in order to form a crystalline structure. The next day the residue is filtered and washed in ether and tetrahydrofuran, and in bidistilled water afterwards. Then the sample is heated in the vacuum-dessicator together with phosphorus pentoxide and paraffin wax till a constant mass is received. As a result 4,02 g. (85,9%) of silver (I) tetra (triphenylarsine) perchlorate is obtained, which melting point is 168-169 ^{0}C. Composition of the deposit is as following: As – 20.54%; Ag – 7.98%; Cl - 2.68% (calculated (%): As – 20,95; Ag – 7,54, Cl – 2,48).

Synthesis of the complex compound of mercury (II) chloride with tri-m-tolylarsine. While stirring, 2.56 g. (0.0074 mol) of etheric solution of tri-m-tolylarsine is added to 2 g. (0.0074 mol) of saturated etheric solution of mercury (II) chloride and stilled at room temperature. The next day the residue is filtered and washed in ether. Then the residue is heated in the vacuum-dessicator together with phosphorus pentoxide and paraffin wax till a constant mass is received. As a result 3.62 g. (0.0058 mol) of a substance with crystalline structure is obtained ($C_{21}H_{21}AsHgCl_2$). The melting point of the substance is 173-174^{0}C. Composition of the deposit is as following: As – 11.70%; Cl – 11.52% (calculated (%): As – 12.09; Cl – 11.45)

Number of complex compounds were obtained using the same methodology as a result of interaction of silver (I) perchlorate and mercury (II) halides with triaryl- and diarylalkyl arsines.

The yield and some physical and chemical characteristics of the synthesized complexes are given in the Table 1 and Table 2.

Results and Discussion

In the work [7] it was indicated that triaryl phosphines are applied as ligands for synthesing complex compounds with silver (I) perchlorates. The application of tertiary arsines for the same purpose has not been investigated so far.

Silver (I) perchlorate does not interacts with symmetrical trialkyl arsines neither at room temperature nor at boiling point of the reacting:

$R_3As + AgClO_4$ X

where R= n–C_4H_9, izo–C_4H_9 or n–C_5H_{12}.

Besides, it appeared that symmetrical trialkyl arsines does not produce crystalline substance as a result of reaction with mercury (II) chloride:

$R_3As + HgCl_2$ X $R_3As – HgCl_2$

where R= n–C_3H_7, izo–C_5H_1 or n–C_7H_{15}.

The reaction is conducted at room temperature. After a significant delay (20-30 days) an oily substance is formed on the bottom of reactor, which neither solve in organic solvents (benzene, ethanol, toluene, n-octane) nor crystallize. One of the goals of this study was the determination of chemical composition of those oily substances without purification, which appeared to be hard to do. While processing products of interaction for their purification purposes, the proportion between ligand and complexing matter dramatically changes.

Experimental data [8] revealed that Ar_2AsR are assimetrical tertiary arsines, where Ar - aryl, and R – alkyl. Besides, they change within a wide range and interact with mercury (II) chloride:

$Ar_2AsR + HgCl_2$ $Ar_2(R)As \cdot HgCl_2$

In order to obtain data related to behavior of tertiary arsines (as ligands), we studied the interaction of triaryl (diarylalkyl) arsines with mercury (II) halides as a result of which the important information related to the composition and properties of the substances under study were received.

The end product is deposited as soon as solutions of silver (I) perchlorate and triphenyl arsine are mixed. In its turn it indicates that the substitution of alkyl radical with aryl radical significantly increases for tertiary arsines the ability to enter into a reaction. The compounds are also obtained as a result of interaction of silver (I) perchlorate with arylalkyl arsines; however, the reaction rate in this case is significantly decreases:

$$AgClO_4 + 4Ar_3As \rightarrow [AsAr_3)_4]ClO_4$$

$$AgClO_4 + 4Ar_2AsR \rightarrow [Ar_2AsR)_4]ClO_4$$

The synthesized substances appeared to be silver (I) tetra (triaryl- and diarylalkyl) arsine perchlorate. The same way, the crystalline substances are obtained as a result of interaction of mercury (II) halides with symmetrical triaryl arsines:

$$Ar_3As + HgX_2 \rightarrow Ar_3As \cdot HgX_2$$

$Ar = C_6H_5$ or m–$CH_3C_6H_4$; and $X = Cl$ or Br.

The reactions between mercury (II) chloride and bromide with diaryl (alkyl) arsines were conducted in etheric solution.

The reactions were conducted applying different concentration and ratio of initial reagents, under heating and cooling conditions in various organic solvents (as tertiary arsines are insoluble in water). The application of tetrahydrofuran appeared to be the most successful; as a result a white coarse-crystalline substance was obtained. It was established that changing the quantitative ratio of reagents within a wide range (1:4 and vice versa) during interaction between tertiary triaryl arsine and mercury (II) chloride and bromide does not influence the chemical composition of the end products: in all cases the end products have a same ratio of 1:1; the identity of the synthesized compounds can be proved by the fact that the mixing of samples obtained at different quantitative ratio, does not change the melting point of the substance. Besides, the best result is reached while using absolute diethyl ether. As for concentration of the end products in unsaturated solution, the reaction rate is significantly low (1-2 months); therefore, on the base of numerous experimental data the following methodology of obtaining the end products was developed: complex compounds are deposited at room temperature in case of interaction of saturated etheric solution of mercury (II) halides with related tertiary arsines taken at molar ratio 1:1; in case of interaction of silver (I) perchlorate with tertiary arsines taken at molar ratio of 1:4.

The synthesized products represent the white coarse-cristalline substances, which are stable while being kept at room temperature, well soluble in dimethylformamide and dimethyl sulfoxide, not as well in aceton and nitromethane, practically isoluble in water, ether, benzene and other nonpolar solvents.

According to the Table 2, the yield of complexes of mercury (II) halides with triaryl arsines is significantly less in comparison with the corresponding diarylalkyl arsines. At the same time the reaction rates of interaction of mercury (II) halides with triphenylarsine are higher than in case of tri-m-tolylarsines, which may be conditioned by factor of sterility and substitutive electronegativity of benzene nucleus in meta-state. It should be indicated that $(m\text{-}CH_3C_6H_4)_3As\cdot HgX_2$ represents more coarse-crystalline substance then other synthesized compounds (influence of the reaction rate).

As it is known [9], mercury (II) halides are hardly ionized in aqueous solutions, as well as in organic solvents, and the concentration of Hg^{2+} ions in saturated solution (e.g. saturated solution of corrosive sublimate) does not exceed 10^{-8}. It indicates the last being a nonelectrolyte $\alpha HgCl_2 < \alpha H_2O$ and in case $HgCl_2$ undergoes electrolytic dissociation it finishes at the first stage:

$$HgCl_2 \qquad HgCl + C^-$$

Table 1
Melting point, yield, molar conductivity (μ) and elemental analysis of silver (I) tetra (triaryl- and diarylalkyl) arsine perchlorates

#	Ar	R	Melting point, $t\ ^0C$	Yield, %	Molar conductivity in DMF at $t=25^0C$, $Om^{-1}\cdot cm^2\cdot mol^{-1}$	Found, %			Molecular formula	Calculated, %		
						As	Ag	Cl		As	Ag	Cl
1	C_6H_5	C_6H_5	168-169	85.9	63.8	20.54	7.98	2.68	$C_{72}H_{60}AgAs_4ClO_4$	20.95	7.54	2.48
2	$m\text{-}CH_3C_6H_4$	$m\text{-}CH_3C_6H_4$	154-155	78.1	69.4	18.86	7.18	2.42	$C_{84}H_{84}AgAs_4ClO_4$	18.76	5.75	2.22
3	C_6H_5	C_2H_5	127-128	79.4	64.2	24.52	8.96	2.96	$C_{56}H_{60}AgAs_4ClO_4$	24.20	8.71	2.86
4	C_6H_5	izo–C_3H_7	139	78,9	62,7	23,75	8,82	2,98	$C_{60}H_{68}AgAs_4ClO_4$	23,16	8,34	2,74
5	C_6H_5	n–C_3H_7	144-145	75,8	67,3	23,48	8,84	2,95	$C_{60}H_{68}AgAs_4ClO_4$	23,16	8,34	2,74
6	C_6H_5	izo–C_4H_9	116-117	78,0	70,8	22,34	8,28	2,84	$C_{64}H_{76}AgAs_4ClO_4$	22,19	7,99	2,63
7	C_6H_5	n–C_4H_9	135-136	76,8	65,2	22,42	8,24	2,81	$C_{64}H_{76}AgAs_4ClO_4$	22,20	7,99	2,63

Table 2
Melting point, yield, molar conductivity (μ) and elemental analysis of complexes of mercury (I) halides with some tertiary arsines

#	Ar	R	Hlg	Melting point, $t\ ^0C$	Yield, %	Molar conductivity in DMF at $t=25^0C$, $Om^{-1}\cdot cm^2\cdot mol^{-1}$	Found, %		Molecular formula	Calculated, %	
							As	Hlg		As	Hlg
1	$m\text{-}CH_3C_6H_4$	$m\text{-}CH_3C_6H_4$	Cl	173-174	79.4	24.6	11.70	11.52	$C_{21}H_{21}AsHgCl_2$	12.09	11.45
2	$m\text{-}CH_3C_6H_4$	C_2H_5	Cl	137-139	82.8	25.7	13.15	13.64	$C_{16}H_{19}AsHgCl_2$	13.44	12.72
3	$m\text{-}CH_3C_6H_4$	izo–C_4H_9	Cl	143-144	87.6	25.4	12.45	12.02	$C_{18}H_{23}AsHgCl_2$	12.79	12.12
4	C_6H_5	n–C_4H_9	Cl	122-123	91.7	23.1	13.40	12.72	$C_{16}H_{19}AsHgCl_2$	13.44	12.72
5	C_6H_5	izo–C_3H_7	Cl	150-151	92.8	28.4	13.28	13.10	$C_{15}H_{17}AsHgCl_2$	13.79	13.05
6	C_6H_5	C_6H_5	Br	180-181	79.4	22.6	11.17	23.89	$C_{18}H_{15}AsHgBr_2$	11.24	23.99
7	$m\text{-}CH_3C_6H_4$	$m\text{-}CH_3C_6H_4$	Br	164-165	76.2	25.4	10.20	22.33	$C_{21}H_{21}AsHgBr_2$	10.57	22.57
8	$m\text{-}CH_3C_6H_4$	C_2H_5	Br	131-139	89.2	27.7	11.49	24.72	$C_{16}H_{19}AsHgBr_2$	11.59	24.73
9	C_6H_5	n–C_4H_9	Br	147-148	82.3	22.4	11.32	24.66	$C_{16}H_{19}AsHgBr_2$	11.59	24.73
10	$m\text{-}CH_3C_6H_4$	izo–C_4H_9	Br	145-146	78.6	26.9	11.09	23.42	$C_{18}H_{15}AsHgBr_2$	11.11	23.70
11	C_6H_5	izo–C_3H_7	Br	158-159	83.1	29.5	11.36	24.98	$C_{15}H_{17}AsHgBr_2$	11.84	25.28

Considering the abovementioned, the bonding of triaryl and diarylalkyl arsines with mercury (II) halides can be explained by nucleophilic mechanism of the quaternisation of arsine, as a result of which cationic complexes are to be produced:

$$Ar_3As + HgHlg_2 \xrightarrow[X]{} [Ar_3As \rightarrow HgHlg]Hlg$$

However, such a conclusion contradicts to the results of physicochemical analysis. Apart from elemental analysis the composition and structure of synthesized substances were studied using physicochemical methods.

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Coordination formulas were established by measuring molar conductivity of the synthesized compounds solved in DMF (dimethylformamide). According to the Table 1, the molar conductivity of silver (I) tetra(triaryl) and tetradiaryl(alkyl) arsine perchlorates ranges within 62-71 Om^{-1}·cm^{2}·mol^{-1} which corresponds to molar conductivity of double-ion type electrolytes[10]. Thereby, it could be concluded that synthesized substances represent cationic complexes, which dissociate in the solution of dimethylformamide according to the following formula:

$$[Ag(AsAr_3)_4]ClO_4 \rightleftarrows [Ag(AsAr_3)_4]^{+} + ClO_4$$

The molar conductivity of complexes of mercury (II) halides with diarylalkyl arsines in unsaturated solutions was studied as well [10]. For substances which contain complex ion and single-charged cations and anions, a definite correlation was revealed between the quantity of ions and conductivity. As these substances, unlike complexes of silver (I) perchlorate with tertiary arsines, does not dissolve in water and their conductivity was determined on the basis of molar conductivity measured in DMF, it could be concluded that the studied complexes are nonelectrolytes. Conductivity of double-ion type complexes in DMF ranges between 60-90 Om^{-1}·cm^{2}·mol^{-1} [10], whereas in our case it does not exceed 30 Om^{-1}·cm^{2}·mol^{-1} (Table 2). Consequently, the synthesized complexes could not be considered as arsenium salts.

In order to investigate the synthesized complexes more thoroughly the IR spectroscopy was applied. Received spectrograms mainly coinside with those of triaryl- and diarylalkylarsines. IR spectrograms of complexes of silver (I) perchlorate with triaryl- and diarylalkyl arsines show absorption bands at 700, 740, 1580, 3000-3080 cm^{-1}, conditioned by vibration of aryl groups (Fig. 1). However, the absorption band of As–C$_{alif}$ bond is biased from 580 cm^{-1} to 625 cm^{-1}, which definitely indicates the quaternization of arsenic atom, the same way it takes place during synthesis of arsonium salts of alkylarines of tertiary arsines [11]. In its turn it proves that ligand is bonded to the central complexing ion Ag^{+} by menas of the loan pair of electrons of 4S^{2} subshell of arsenic atom.

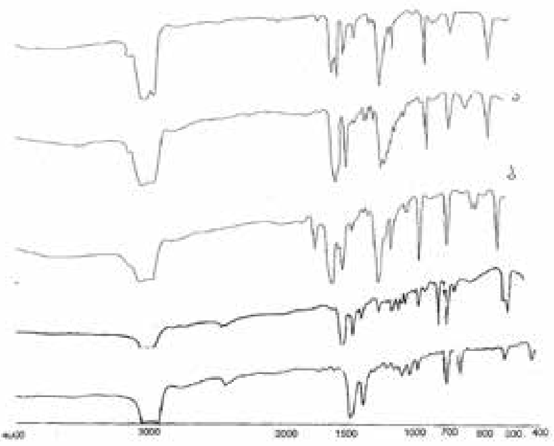


Figure 1. IR absorption spectra
1. $[Ag(As(C_6H_5)_3)_4]ClO_4$;2. $[Ag(As(C_3H_7\text{-izo})(C_6H_5)_2)_4]ClO_4$ 3.$[Ag(As(C_6H_4CH_3\text{-}m)_3)_4]ClO_4$; 4. $HgCl_2(C_6H_5)_2AsC_4H_9$–n

5. $HgBr_2$(m - $CH_3C_6H_4)_3AsC_4H_9$–izo

The analysis of spectrograms of complexes of mercury (II) chlorides and bromides with tertiary arsines revealed the absence of absorption bands of As–C$_{alif}$ bond at 560-580 cm^{-1}. However, the absorption band appears at 620 cm^{-1}, which is characteristic for As–C$_{alif}$ bond, when arsenic atom is in SP^{3} hybridization state.

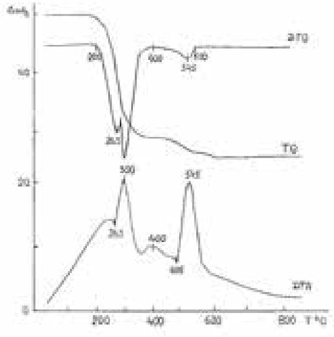
Considering the measured molar conductivity and results of IR spectroscopy it may be concluded that the studied substances represent bridged dimeric complexes.

The thermal analysis was applied in order to study physical and mechanical properties of synthesized complexes. A derivatogram of silver (I) perchlorate with tetra (triphenyl) arsine provided as an example (as thermolysis of all substances carry on nearly the same way) (Fig. 2). Thermolysis comprises of four endothermal effects and three exothermal effects. The sample starts loosing the mass at 200^{0}α. Within the temperature interval of 200–400^{0}α the mass is reduced by 82.4% (theoretical – 85.55%), which corresponds to the detachment of the ligand (organic part of the complex). Within this range, namely at 300^{0}α the exothermal effect takes place; however, no increase in mass is observed. The following reduction of the mass by 7.8% (theoretical – 7.0%) takes place at temperature interval of 400–550^{0}α and corresponds to the lost of anion. As ClO_4 does not exist, it can be concluded that the oxygen and chlorine dioxide are detached, which is proved by endothermal effects at 470^{0}α and 495^{0}α. As for exothermal effects at 300^{0}α, 400^{0}α and 545^{0}α, it may be presumed that they are conditioned by "intramolecular rearrangement". Considering the abovementioned, the thermolysis of the substance may be presented as follows:

$$[Ag(AsAr_3)_4]ClO_4 \rightleftarrows [Ag(AsAr_3)_4]^{+} + ClO_4$$

The same processes are observed during the thermolysis of other synthesized substances. Today the intensive studies are conducted on investigating physiological activity of the synthesized complexes.

Thereby, as a result of interaction of triaryl- and diarylalkyl arsines with silver (I) perchlorate complexes of silver (I) tetra (triaryl- and diarylalkylarisno) perchlorates with coordination number 4 were synthesized. Based on the results of thermogravimetric analysis the possible scheme for ther-



molysis of complex compounds is provided.

Figure2.

Thermogravigram of

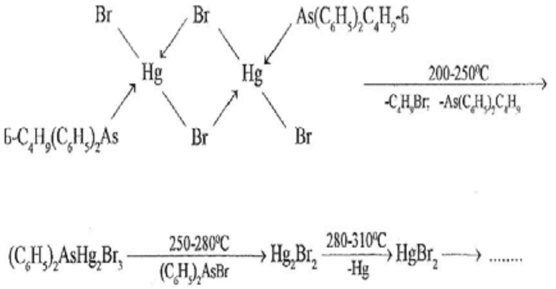
$[Ag(As(C_6H_5)_3As)_4]ClO_4$

The different character of thermolysis of complexes of mercury (II) halides with tertiary arsines was proved experimentally. The thermolysis of all samples progress the same way as the composition and structure of synthesized complexes are similar. The problem was that the decomposition temperature of ligands does not differ much from that of mercury (II) halides; however, the certain regularities have been established. As an example a derivatogram of the complex of mercury (II) bromide with diphenyl-n-butylarsine is provided.

As the derivatogram shows the thermolysis comprises of several stages reflecting three endothermal effects and one exothermal effect. The first endothermal effect is observed at 110–115^{0}C, which is not accompanied by the loss of mass. As this temperature range does not corresponds to the melting point of the ligand (147-148^{0}C), it can be presumed that the "intermolecular rearrangement" takes place., The mass loss starts at 200^{0}C and continues till 400–500^{0}C. The mass is decreased by 33.8% at the range of 200-250^{0}C, which corresponds to the loss of butylbromide C_4H_9Br and diphenylbutylarsine $(C_6H_5)_2AsC_4H_9$ (32,6%); however, in the last case the endothermal effect is not observed. The second endothermal effect at the range of 250-280^{0}C with its peak at 270^{0}C, corresponds to the loss of a molecule of diphenylbromarsine (mass loss - 24.8% (theoretical – 23.9%)). The following exothermal effect is conditioned by the changes in molecular structure as a result of loss of diphenylbromarsine. The last endothermal effect at the interval of 280–310^{0}C, with its peak at 300^{0}C corresponds to loss of one molecule of mercury from Hg_2Br_2 (mass loss – 16.7% (theoretical – 15.5%)).

The comparison of DTA, DTG and TG curves allows to presume that within interval of 200–310^{0}C whole "organic part" as well as the part of complex forming agent are detached, which are definitely endothermal processes.

Considering the abovementioned the thermolysis of the studied sample may be represented by the following scheme:



Hereby, the dimeric bridged complexes are synthesized as a result of interaction of mercury (II) halides with triaryl- and diarylalkyl arsines.

Roentgenographic analysis of synthesized coordination compounds was conducted. The analysis revealed that synthesized complexes represent crystalline substances and are characterized by the individual generality of relative intensity and interplanar spacing.

4. Conclusions

The interaction of silver (I) perchlorate and mercury (II) halides with triaryl- and diarylalkyl arsines was studied. As a result the complex compounds were obtained having the following formulas

$[Ag((C_6H_5)_2R)_4]ClO_4$ and $HgHlg_2\cdot As(R)Ar_2$, where $Ar=-C_6H_5$, $m-CH_3C_6H_4$; $R=-C_6H_5$, $m-CH_3-C_6H_4$, C_2H_5, $izo-C_3H_7$, $n-C_3H_7$, $izo-C_4H_9$.

and , where ; , , , , , . Both the elemental and physicochemical analysis were applied to study composition and structure of the synthesized compounds.

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