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Synthesis and characterization of aspartic acid complexes of antimony and bismuth triiodide

Short Communication

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Summary. New bioinorganic complexes of the aspartic acid with the antimony or bismuth triiodide were synthesized by a direct solid–solid reaction at room temperature. The formula of the complex is $MI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 2.5H_2O$ ($M = Sb$, Bi). The complex may be a dimer with bridge structure. The crystal structure of the complexes belongs to a triclinic system. The lattice parameters are $a = 0.9883$ nm, $b = 1.4284$ nm, $c = 2.0114$ nm, $\alpha = 94.46°$, $\beta = 99.76°$ and $\gamma = 100.1°$ for the complex of antimony and $a = 0.9756$ nm, $b = 1.4560$ nm, $c = 1.9875$ nm, $\alpha = 94.18°$, $\beta = 97.25°$ and $\gamma = 101.16°$ for the complex of bismuth. The infrared spectra and thermal analyses can demonstrate the complex formation between the aspartic acid and the antimony or bismuth ion.

Keywords: Aspartic acid complex of Sb or Bi – Solid solid reaction synthesis – Characterization

Introduction

Although the main group metal elements, such as arsenic, antimony and bismuth, possess rather toxicity, the main group elements can also have a certain medical function (Cantos, 1993). Some compounds of arsenic, antimony and bismuth are often used as the chemical medicaments. Especially, some of the compounds can have antitumer activity (Kopf-Maier, 1988). Pharmacological applications of the inorganic complexes composed of the metal ion and organic ligands have also been reported (Bakhtiar, 1999). Still, the bioinorganic complexes of the main group metal elements have been rare. There are always the natural amino acids in the human body. Therefore, to study if or how the bioinorganic complexes between the various natural amino acids and the main group metal elements can be formed will be very interesting not only for the biologic chemistry of main group metal elements, but also for the medicinal application of the amino acid complexes. Aspartic acid is one of the natural amino acids in the human body. We find that the aspartic acid complex of antimony or bismuth triiodide can be synthesized very easily through a direct solid–solid reaction of the aspartic acid and antimony or bismuth triiodide powder. Here, we report the preparation process and characterization of the bioinorganic complexes of aspartic acid with antimony or bismuth triiodide.

Methods

The aspartic acid is levorotatory stereoisomer. The preparation of the complex of aspartic acid and antimony triiodide: First, to weigh 1.006 $HOOCCH_2CH(NH_2)COOH$ and 1.510 g SbI_3 and very well to mix up the two reactants together in an agate mortar. The molar ratio of SbI_3 to $HOOCCH_2CH(NH_2)COOH$ is 1:2.5. Then, carefully grind the mixture. At the first, the color of the mixture was brown, then, became orange. This indicated that the reaction did happen. The reaction conducted in grinding at room temperature lasted for about 6 h. Last, the resultant was dried in a vacuum drying oven for 4 h. The resultant is the orange powder. The resultant is stable in air and does not show appreciable moisture. The preparation of the complex of aspartic acid and bismuth triiodide: To weigh 1.006 g $HOOCCH_2CH(NH_2)COOH$ and 1.762 g BiI_3 and very well to mix up the two reactants together in an agate mortar. The molar ratio of BiI_3 to $HOOCCH_2CH(NH_2)COOH$ is also 1:2.5. The black powder resultant can be obtained by the similar procedure. The resultant is also stable in air and shows no appreciable moisture.

The powder x-ray diffraction pattern of the resultants was recorded by a D/max-YB x-ray diffractometer, $CuK_{\alpha 1}$ radiation, scanning rate $2(2\theta)$/min, at room temperature. The results of indexing to the powder x-ray diffraction patterns of the resultants are listed in Tables 1 and 2, respectively. The infrared spectra of the resultants were also measured by a

Table 1. The experimental data and the calculated results for powder x-ray diffraction pattern of $SbI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 2.5H_2O$ triclinic system: a = 0.9883 nm, b = 1.4284 nm, c = 2.0114 nm
$\alpha = 94.46°$, $\beta = 99.76°$, $\gamma = 100.1°$

h, k, l	dexp (nm)	dcal (nm)	I/I_0	h, k, l	dexp (nm)	dcal (nm)	I/I_0
0 2 0	0.6986	0.6988	7.7	1 −3 7	0.2413	0.2414	7.3
1 −1 −2	0.6868	0.6877	4.0	3 1 4	0.2354	0.2354	2.0
0 1 −3	0.6213	0.6214	1.9	2 −5 4	0.2332	0.2331	1.8
1 −1 −3	0.5527	0.5518	6.5	3 0 5	0.2280	0.2278	3.0
0 2 −3	0.5069	0.5074	1.6	2 −4 6	0.2234	0.2236	3.9
1 −2 −3	0.4576	0.4576	4.3	2 1 7	0.2162	0.2160	26.1
2 −1 −3	0.4211	0.4194	3.2	2 5 −5	0.2108	0.2108	2.7
1 3 −2	0.4103	0.4097	12.5	2 −4 7	0.2067	0.2067	1.9
1 3 0	0.3900	0.3903	4.4	0 7 −3	0.1975	0.1972	13.2
1 2 3	0.3751	0.3750	5.4	3 4 3	0.1944	0.1941	3.3
2 2 −3	0.3490	0.3488	18.1	5 −4 −2	0.1837	0.1841	7.0
1 −2 −5	0.3368	0.3367	3.1	0 6 6	0.1808	0.1808	10.1
2 −1 −5	0.3301	0.3301	100.0	2 7 −3	0.1744	0.1743	1.7
2 −1 4	0.3210	0.3211	13.8	4 2 6	0.1649	0.1647	5.3
3 −2 −1	0.3164	0.3163	9.5	3 7 −1	0.1581	0.1580	1.7
0 1 6	0.3120	0.3120	10.7	2 8 3	0.1458	0.1456	3.1
3 1 −2	0.3021	0.3028	5.0	4 6 4	0.1386	0.1386	5.4
3 0 2	0.2875	0.2878	5.6	−5 −4 −5	0.1357	0.1357	4.1
0 −5 2	0.2774	0.2771	2.1	4 8 −1	0.1311	0.1310	2.7
2 4 0	0.2591	0.2594	2.9	4 6 7	0.1248	0.1246	1.9
0 3 6	0.2553	0.2554	31.5	−5 −6 −5	0.1209	0.1211	2.3

Table 2. The experimental data and the calculated results for powder x-ray diffraction pattern of $BiI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 2.5H_2O$ triclinic system: a = 0.9756 nm, b = 1.4560 nm, c = 1.9875 nm
$\alpha = 94.18°$, $\beta = 97.25°$, $\gamma = 101.16°$

h, k, l	dexp (nm)	dcal (nm)	I/I_0	h, k, l	dexp (nm)	dcal (nm)	I/I_0
0 2 −1	0.6689	0.6906	25.8	1 1 8	0.2219	0.2217	1.1
0 1 −3	0.6197	0.6180	4.2	1 6 −3	0.2168	0.2165	34.5
0 2 2	0.5520	0.5500	3.4	4 −4 2	0.2109	0.2106	0.3
1 0 3	0.5046	0.5055	1.2	3 2 5	0.2069	0.2070	5.1
2 −1 −3	0.4088	0.4094	10.4	2 6 −3	0.1962	0.1960	14.1
0 1 −5	0.3887	0.3883	2.8	3 5 2	0.1836	0.1837	2.8
1 −3 −3	0.3741	0.3733	5.0	0 7 4	0.1812	0.1813	11.9
0 4 −2	0.3447	0.3453	7.7	4 −1 7	0.1726	0.1725	5.6
1 2 −5	0.3396	0.3389	1.8	0 7 6	0.1650	0.1652	7.4
0 2 5	0.3297	0.3298	100.0	2 8 −2	0.1578	0.1576	4.2
2 −3 −3	0.3212	0.3211	2.1	2 7 6	0.1455	0.1456	3.5
2 −2 4	0.3159	0.3157	4.2	7 −2 −3	0.1391	0.1390	7.6
1 4 2	0.2869	0.2866	3.9	4 6 5	0.1351	0.1353	8.1
3 −2 −4	0.2769	0.2768	0.7	4 8 −3	0.1313	0.1313	4.6
3 2 −4	0.2539	0.2536	39.5	4 8 −5	0.1295	0.1294	2.5
3 3 −1	0.2439	0.2441	1.4	5 5 6	0.1252	0.1251	3.0
3 2 −5	0.2407	0.2405	3.3	5 5 7	0.1208	0.1209	2.5
1 5 3	0.2300	0.2301	4.4				

Nicolet 5SXC spectrometer and the potassium bromide disk technique. The infrared spectra of the resultants are shown in Fig. 1. Thermal analyses of the resultants were performed by a LCT-1 differential thermal balance in air, a heating rate of $10°C\,min^{-1}$ and the α-Al_2O_3 reference. The sample mass was 18.4 mg and 18.8 mg for the aspartic acid complex of antimony and bismuth, respectively. The possible pyrolysis reactions in the thermal decomposition processes of the complexes, the experimental and calculated results are summarized in Table 3.

Results and discussion

All the diffraction peaks in the pattern of the each resultant can be readily indexed by a set of lattice parameters according to triclinic system, although the number of the diffraction peaks in the pattern is more. Tables 1 and 2

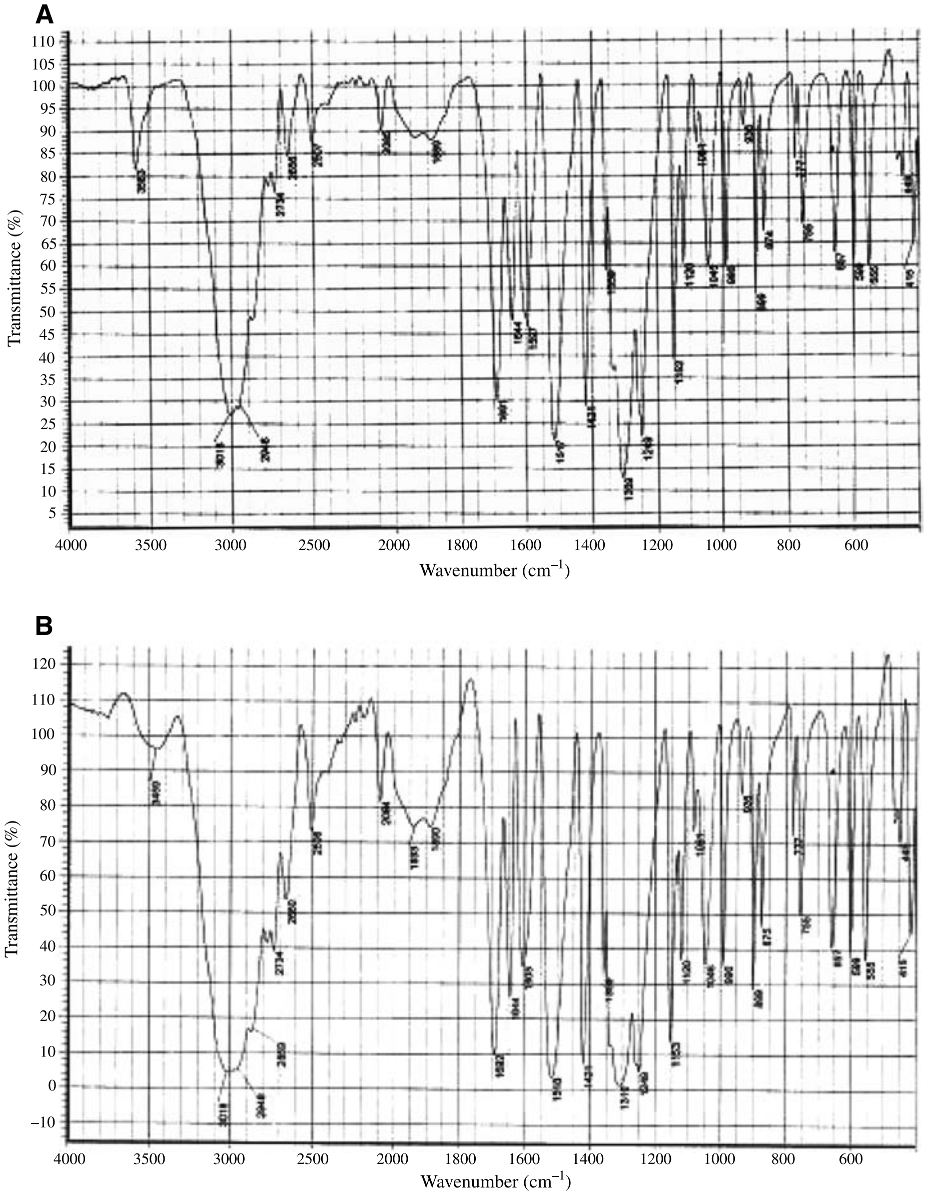

Fig. 1. The infrared spectra of $SbI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 2.5H_2O$ and $BiI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 2.5H_2O$. **A** $SbI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 2.5H_2O$; **B** $BiI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 2.5H_2O$

show that all relative deviations between the calculated and experimental d_{hkl} are less than 0.5%. This indicates that the resultant is a single-phase compound with triclinic symmetry. The crystal structure of antimony and bismuth triiodide belongs to hexagonal system (JCPDS7-273 and 7-269) and that of L-aspartic acid belongs to monoclinic system (JCPDS 23-1519). The crystal structure of the resultants belongs neither to the hexagonal nor to monoclinic system, but to a triclinic system. Obviously, the resultants must be the new complexes with the formula $MI_3[HOOCCH_2CH(NH_2)COOH]_{2.5}$ (M = Sb, Bi). Many complexes of antimony and bismuth are a dimer (Greenwood, 1997). Therefore, we can not rule out that the complexes may also be a dimer. The configuration of the complex dimer will possess a bridge structure. If this be so, the possible formula of the complex will be $I_3M[HOOCCH_2CH(NH_2)COOH]_5MI_3$. We find that the solid complex with the 1:3 molar ratio of SbI_3 to $HOOCCH_2CH(NH_2)COOH$ can not be prepared by the similar method.

Figure 1 shows the infrared spectra of the resultants. At first glance, the infrared spectra of the complexes seem to

Table 3. Thermal decomposition data of $SbI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 2.5H_2O$ and $BiI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 2.5H_2O$

Reaction	T (°C)	Total weight loss (%)	
		$W_{exp.}$	$W_{theor.}$
$SbI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 2.5H_2O$			
$\downarrow -H_2O$	180 (endo.)	2.1	2.15
$SbI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 1.5H_2O$			
$\downarrow -1.5H_2O, -NH$	210 (endo.)	4.7	5.03
$SbI_3[OOCCH_2CH_2CO][OOCCH_2CH(NH_2)CO]_{1.5}$			
$\downarrow -2I,$			
$\downarrow -1.5[OOCCH_2CH(NH_2)CO]$	240 (endo.)	49.1	50.02
$SbI[OOCCH_2CH_2CO]$			
$\downarrow -OCCH_2CH_2CO$	320 (exo.)	11.6	10.05
SbIO			
$\downarrow -I$	420	14.0	15.19
0.5OSbSbO			
$\downarrow +0.25O_2$	550 (exo.)		
$0.5Sb_2O_3$		18.5*	17.56**
$BiI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 2.5H_2O$			
$\downarrow -H_2O$	210 (endo.)	2.1	1.95
$BiI_3[OOCCH_2CH(NH_2)CO]_{2.5} \cdot 1.5H_2O$			
$\downarrow -1.5H_2O$			
$\downarrow -0.5[OOCCH_2CH(NH_2)CO]$	240 (endo.)	9.9	9.16
$BiI_3[OOCCH_2CH(NH_2)CO]_2$			
$\downarrow -2I$			
$\downarrow -OOCCH_2CH(NH_2)CO$	350 (exo.)	49.5	50.71
$\downarrow -OCCH_2CH(NH_2)CO$			
BiIO			
$\downarrow -I$	400	13.0	13.76
0.5OBiBiO			
$\downarrow +0.25O_2$	510 (exo.)		
$0.5Bi_2O_3$		25.5*	24.42**

* The percentage weight of the residue in the sample

** The percentage content of the metal oxide in the complex

be similar to that of aspartic acid (Fluca, 1974). However, the intensities of some absorption peaks change, especially, the new absorption peaks appear in the infrared spectra of the complexes. Unlike in the infrared spectrum of aspartic acid, there is an appreciable absorption peak at $3459\,cm^{-1}$ or $3583\,cm^{-1}$ in the infrared spectrum of the complex. Generally, the absorption peak at about $3500\,cm^{-1}$ in the infrared spectra can be assigned to the stretching vibration of the O–H bond from the water molecule (Nakanshi, 1977). This indicates that there may be the water molecule in the solid complexes. Because the synthesis reaction is a solid–solid reaction without any aqueous solution, the water molecules in the solid complex must be produced in the reaction process. Therefore, we can assume that the dehydration of the aspartic acid may occur in the synthesis process of the complex, that is, the two carboxylic groups of the aspartic acid molecule will lose a water molecule and become the acid radical group (–CO–O–OC–). Then, the oxygen atom of the acid radical group (–CO–O–OC–) can directly coordinate to the metal atom to form the complex. The water molecules in the complex will exist as the lattice water molecules in the lattice of the solid complex. Perhaps, it is just why the absorption peak from the stretching vibration of the O–H bond in the water molecule can appear in the infrared spectra of the complexes. If there is the acid radical group (–CO–O–OC–) in the solid complex, we can reasonably expect that there must also be the corresponding absorption peaks from the various vibrations of the –CO–O–OC– group in the infrared spectra of the complex. Indeed, there is an appreciable absorption peak at $1890\,cm^{-1}$ or $1889\,cm^{-1}$ in the infrared spectrum of the complex of antimony or bismuth. This absorption peak is characteristic for the –CO–O–OC– group in the infrared spectrum (Nakanshi, 1977). However, there is no such absorption peak at about $1890\,cm^{-1}$ in the infrared spectrum of aspartic acid (Fluca, 1974). Besides, generally, the absorption peak from the skeleton vibration of the C–O–C link in the acid radical group (–CO–O–OC–) is always at about 1300 cm^{-1} in the infrared spectra of the carboxylic acid radicals (Nakanshi, 1977). We have noted that the absorption peak at 1510 cm^{-1} is the most intense peak in the infrared spectrum of aspartic acid (Fluca, 1974). However, the most intense absorption peak is at 1310 cm^{-1} or 1309 cm^{-1} in the infrared spectrum of the complex of antimony or bismuth. We consider that it is due to the –CO–O–OC– group in the solid complexes that the absorption peak at 1310 cm^{-1} or 1309 cm^{-1} from the skeleton vibration of the C–O–C link in the CO–O–OC– group will become such intense. The free amino acid often exists as an inner salt. There are always both protonated amino group ($-NH_3^+$) and deprotonated carboxyl group ($-COO^-$) in the free amino acid. But, due to the dehydration of the aspartic acid and the formation of the –CO–O–OC– group in the synthesis process of the complex, there may be only the amino group ($-NH_2$), not any protonated amino group ($-NH_3^+$) in the solid complex. The effective ionic radius (0.076 nm and 0.103 nm) of the Sb^{3+} and Bi^{3+} ion is larger and the coordination number of the Sb^{3+} and Bi^{3+} ion can also be six or eight (Shannon, 1976; Wullens, 1996). Therefore, the Sb^{3+} or Bi^{3+} ion in the solid complex can be coordinated not only by the iodine atom, but also by the oxygen atom of the acid radical group (–CO–O–OC–) or the nitrogen atom of the amino group ($-NH_2$). If the complex molecule is a dimer with the bridge structure, the two oxygen atoms of the acid radical groups (–CO–O–OC–) can bond to different Sb^{3+} (or Bi^{3+}) ions, respectively. Hence, the bridge bonding between the two Sb^{3+} (or Bi^{3+}) ions can be formed in the solid complex. Perhaps, the possible configuration of the complex dimer will be $\{[OOCCH_2CH(NH_2)CO]_2I_3M[OOCCH_2CH(NH_2)CO]MI_3[OOCCH_2CH(NH_2)CO]_2\} \cdot 5H_2O$ ($M = Sb$ or Bi). That is, the oxygen atoms of two –CO bonds in one acid radical group (–CO–O–OC–) from the organic ligands are coordinated to two Sb^{3+} (or Bi^{3+}) ions, respectively. So, some M–OC–O–CO–M bridge links may exist in the solid complex. We have noted that the cell volume of the complex crystal is larger. This may also be because the solid complex molecules possess the longer and larger bridge links. Besides, the complex formation between the aspartic acid and the Sb^{3+} or Bi^{3+} ion can also explain why the solid complex does not show appreciable moisture in air, unlike the antimony or bismuth triiodide. This is because all Sb^{3+} or Bi^{3+} ions in the solid complex have been coordinated or surrounded very well by the iodine and oxygen atoms. In this case, any water molecules in air can not close to the Sb^{3+} or Bi^{3+} ion at the center of the inner coordination sphere of the complex. The above conclusions can also be supported by thermal analysis process of the solid complex.

As Table 3 shows, the first mass loss of the Sb complex sample happens at 180°C. The experimental percentage mass loss (2.1%) indicates the loss of one water molecule. The calculated percentage mass loss is 2.15%. The dehydration needs energy, hence, there is an endothermic peak in TDA curve. Higher dehydration temperature means that the water molecules in the solid complex must be the lattice water, but and not be the sorption water from the air. Then, at 210°C, the sample will lose the residual 1.5 water molecules and one amino group ($-NH_2$). The calculated percentage mass loss (5.05%) is very close to the experimental that (4.7%). To check if the pyrolysis product is the amino group, a certain amount (about 20 mg) of the complex is placed in a glass test-tube and slightly heated by a gas lamp. The gaseous pyrolysis product is detected by using the wet pH paper. The results show that the gaseous pyrolysis product can make the wet pH paper become blue. This shows that the pyrolysis product is a basic gas. At about 240°C, the sample will lose 1.5 organic ligands and 2 iodine atoms. The experimental percentage mass loss (49.1%) is close to the calculated that (51.01%). Due to the reaction of the pyrolysis product and the oxygen in air, hence, there is a corresponding exothermic peak in TDA curve. At 320°C, the sample will lose the residual organic ligands. When temperature is higher 400°C, the sample will lose the residual iodine atoms and become OSb–SbO. With temperature increasing, the pyrolysis product OSb–SbO will be oxidized by the oxygen in air and become gradually to Sb_2O_3. Last, the pyrolysis residue is yellow Sb_2O_3 powder. Similarly, the solid complex of aspartic acid and bismuth triiodide will also lose one water molecule, but the dehydration temperature (210°C) of the solid complex of bismuth is slightly higher than that (180°C) of the solid complex of antimony. Then, at 240°C, the sample will lose 0.5 organic ligand and the residual lattice water molecules. The elimination of the organic ligands from the complex and the dehydration of the complex need a certain energy, therefore, there is a corresponding endothermic peak in TDA curve. When temperature is above 300°C, the sample will lose two iodine atoms and the residual organic ligands. The experimental percentage mass loss (49.5%) consists with the calculated that 50.71%. Because the thermal decomposition temperature is higher, the pyrolysis products can react with the oxygen in air, there is a larger exothermic peak in TDA curve. Last, the residue is black Bi_2O_3 powder. Obviously, the thermal analysis process shows that the solid complex prepared by a direct solid–solid reaction of the aspartic acid and antimony or bismuth triiodide powders does contain the lattice water molecules. As mentioned above, the infrared spectra of the complexes have shown that there is the absorption peak from the vibration of the O–H bond of the water molecule in the infrared spectra of the complexes at room temperature. This indicates that the solid complexes have contained some water molecules at room temperature. Therefore, the loss of the water molecules from the solid complex must not be due to the dehydration of the aspartic acid in the thermal decomposition process. It is most likely that the water molecules in the solid complex are produced in the synthesis process of the complex. Especially in the thermal decomposition process of the solid complex, the elimination of the organic ligands from the complex is as the aspartic acid radical groups, not as the aspartic acid molecule. This experimental fact also indicates there are the aspartic acid radical groups in the solid complex. Therefore, the results of thermal analysis of the complexes very well consist with the results of the infrared spectra of the complexes. Besides, we have also noted that the elimination of all the acid radical groups or amino groups from the complex does not occur at one time. The experimental phenomena demonstrate that the bonding or coordination of the oxygen atoms or nitrogen atoms of the aspartic acid radical groups to the Sb^{3+} or Bi^{3+} ion must be different from each other. So, we can assume that the aspartic acid radical groups (–CO–O–OC–) eliminated from the complex at higher temperature may be in the M–OC–O–CO–M bridge links. Because the formation of the M–OC–O–CO–M bridge link can strengthen the bonding between the acid radical group (–CO–O–OC–) and the Sb^{3+} or Bi^{3+} ion. In this case, the elimination of the acid radical groups (–CO–O–OC–) in the M–OC–O–CO–M bridge link from the complex will become more difficult. If so, we can consider that the number of the Bi–OC–O–CO–Bi bridge links in the solid complex of bismuth may be more than the number of the Sb–OC–O–CO–Sb bridge links in the solid complex of antimony. It is very easily understood because the effective ionic radius (0.103 nm) of the Bi^{3+} ion is much larger than that (0.076 nm) of the Sb^{3+} ion (Shannon, 1976), hence, the steric hindrance for the formation of the bridge links in the solid complex of bismuth must be much smaller than that in the solid complex of antimony. Besides, we have also noted that the elimination temperature of three iodine atoms from the solid complex is also different. This indicates that three iodine atoms must possess different chemical environment in the solid complex. That is, the coordination or bonding of three iodine atoms to the Sb^{3+} or Bi^{3+} ion in the solid complex must also be different, unlike in the antimony or bismuth triiodide.

Summing up, the solid complex of the aspartic acid and antimony or bismuth triiodide can be synthesized through a direct solid–solid reaction of the aspartic acid with SbI_3 or BiI_3 powder at room temperature. The complex crystal belongs to the triclinic system. In the synthesis reaction process, first, the aspartic acid will dehydrate, then, coordinated to the Sb^{3+} or Bi^{3+} ion through the oxygen atoms of the acid radical groups (–CO–O–OC–). The two oxygen atoms of some (–CO–O–OC–) groups will bond to different the Sb^{3+} (or Bi^{3+}) ions, respectively. There may be the (M–OC–O–CO–M) bridge links in the solid complex. The complex may be a dimer. The complexes of other amino acids and antimony or bismuth may also be prepared by similar method. Perhaps, this method can be used to synthesize the bioinorganic complexes of other main group metal ions and amino acid or some biologic ligands.

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