ISSN 1070-3632, Russian Journal of General Chemistry, 2008, Vol. 78, No. 10, pp. 1836–1842. © Pleiades Publishing, Ltd., 2008.
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Reaction of a Mixture of Bismuth and Iron Oxides with Chlorine and Sulfur Dioxide

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Received April 10, 2008

Abstract—The processes in a heterogenous multicomponent system Bi_2O_3–Fe_2O_3–Cl_2–SO_2. are explored. In the temperature range 300–700°C is clearly developed mutual influence of chemical reactions at introducing to the system of an additional component: chloridosublimation of both bismuth and iron in the presence of SO_2 and chloridosublimation of bismuth at adding iron oxide to bismuth oxide are accelerated. In the region of the higher temperatures the possible chemical reactions in the system proceed independently: SO_2 only dilutes chlorine and mutual influence of bismuth and iron oxides is not found.

DOI: 10.1134/S1070363208100022

Revealing the optimal conditions for conducting a chemical process in multicomponent heterogenous system widely used in chemical technology meets a problem defined by mutual influence of chemical reactions. Change in the composition of reaction mixture and, in particular, adding to the system of any additional component leads to changes in both equilibrium composition of reaction products and rates of chemical processes [1].

A convenient model for the investigation of mutual influence of chemical reactions in heterogenous systems “gas–condensed phase” is the process of chemical sublimation. In such process a substance is transformed from condensed phase into gas owing to chemical reactions. A particular case of chemical sublimation is the process of chlorination where only gaseous reaction products are formed. In this case the process kinetics is not complicated by diffusion of gaseous components through the film formed by reaction products.

Investigations of chlorination of mixtures of oxides have been carried out earlier mainly for revealing optimal conditions for separating the components [2, 3]. In some works has been shown that certain conclusions about kinetics of chlorination of mixtures can not be achieved from the results obtained for individual metal oxides: the rate of chlorination of Ga_2O_3, In_2O_3 and ZnO increases noticeable in the presence of Fe_2O_3 [4, 5]. Variations in composition of gas phase, in part, dilution of chlorine with sulfur dioxide also leads to changes in the rate of chlorination [6, 7].

In the present work we explored interactions in the system Bi_2O_3–Fe_2O_3–Cl_2–SO_2 for the estimation of variations in the rate of $BiCl_3$ and $FeCl_3$ formation at the complication of the reaction system. Such investigation are interesting not only for revealing regularities of the processes in multicomponent heterogenous systems but also for finding optimal conditions of $BiCl_3$ separation from the mixtures containing Fe_2O_3. The importance of obtaining pure $BiCl_3$ is defined by its wide application in preparative chemistry and in catalysis and because it is used as a basic material for preparation of metallic bismuth [8, 9].

Reaction of Bi_2O_3 with Cl_2 proceeds even at room temperature, the products are bismuth oxychloride BiOCl and oxygen. Chemical sublimation of bismuth trichloride at 25–250°C does not occur. The $BiCl_3$ sublimate appears at 300°C. The chloridosublimation completeness at the temperature below 400°C is only 5% or less (Table 1), the bismuth remains in the solid phase in the form of oxychloride. We suggest possible to conclude that BiOCl is an intermediate at the chemical sublimation of $BiCl_3$, and in the temperature range 25–400°C the rate of BiOCl formation is much higher than the rate of the bismuth chloridosublimation.

The isotherms of interaction of bismuth oxide with chlorine at 500–650°C (Fig. 1) also show that the process of $BiCl_3$ formation consists of separate steps. In the initial moment the sample mass increases due to formation of BiOCl, and then decreases practically lineary in mass due to sublimation of $BiCl_3$. Above 650°C no increase in the initial mass is registered and the process includes induction step whose duration falls when temperature increases. The activation energy of the process of $BiCl_3$ chemical sublimation is 115 kJ mol^{-1}.

Reaction of Bi_2O_3 with SO_2 begins at 450°C and proceeds with increase in the sample mass. By the data of chemical analysis, content of sulfur in the solid phase reaction products achieves 4.0% at the process temperature 500°C and contact duration 5 h. The main portion of sulfur is sulfate, that is, the total process can be represented by the following equation:

$$4\,Bi_2O_3 + 12\,SO_2 = 3\,Bi_2(SO_4)_3 + Bi_2S_3. \quad (1)$$

Under polythermic conditions (heating rate 15 deg/min) the increase in mass occurs until ~650°C, but further increase in temperature leads to decrease in the sample mass, and the content of sulfur in the residue also falls. The apparent activation energy of the process at the contact duration 60 min in the temperature range 450–550°C is 130 kJ mol^{-1}. Formation of new solid phase leads to sharp decrease in the rate of the process, and even after 5 h contact of Bi_2O_3 with SO_2 at 500°C the process deepness is not higher than 20%. Elevating the temperature leads to stepwise decomposition of bismuth sulfate [10], content of sulfur in the solid phase falls and at 900°C becomes equals to 0.9% only.

Reaction of Bi_2O_3 with a mixture of Cl_2 and SO_2 occurs even at room temperature, the reaction products are predominantly bismuth sulfate and oxychloride.

$$4\,Bi_2O_3 + 3\,Cl_2 + 3\,SO_2 = 6\,BiOCl + Bi_2(SO_4)_3. \quad (2)$$

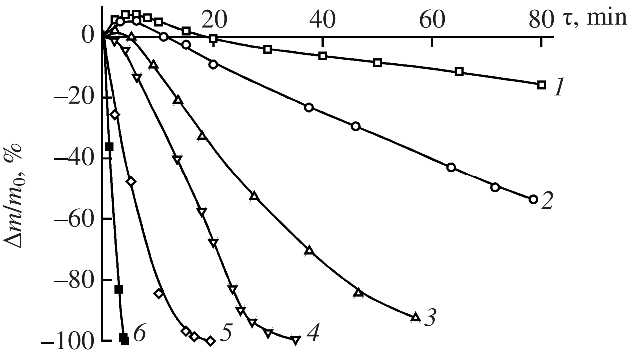

Fig. 1. Isotherms of reaction of Bi_2O_3 with Cl_2. (*1*) 500, (*2*) 550, (*3*) 600, (*4*) 650, (*5*) 700, and (*6*) 750°C.

Table 1. Completeness of chloridosublimation of bismuth and iron at the chlorination of Bi_2O_3 and Fe_2O_3 (% to initial)

t, °C	τ, min	Bi, (% to initial)		Fe, (% to initial)	
		Cl_2	$Cl_2 + SO_2$	Cl_2	$Cl_2 + SO_2$
300	30	1.2	4.4	0.3	19.1
	60	1.5	7.4	0.5	23.0
	90	1.5	10.3	0.5	25.2
350	30	1.6	6.5	0.8	20.0
	60	2.3	8.6	1.0	22.0
	90	2.7	11.8	0.9	26.5
400	30	2.6	7.0	1.0	22.0
	60	3.9	10,6	1.5	23.1
	90	5.2	12.5	1.7	28.0
450	30	8.9	21.8	1.8	22.5
	60	15.7	34.4	2.3	27.3
	90	20.3	46.3	2.4	29.5
500	30	12.5	24.5	3.2	25.6
	60	18.9	38.2	5.4	30.6
550	30	25.9	37.3	3.8	–
	60	47.6	52.5	6.2	–
600	30	58.1	61.0	8.8	31.4
650	30	93.7	82.6	18.8	38.1
700	30	96.6	–	36.6	57.1

Chemical sublimation of $BiCl_3$ in this case occurs at 300°C (Table 1). Completeness of chloridosublimation raises practically lineary in time. The condensed phase consists mainly of BiOCl and $Bi_2(SO_4)_3$ (Table 2). Formation of solid phase products in conjunction with low volatility of $BiCl_3$ vapor decelerate further progress of the process. In the temperature range 300–400°C the fraction of bismuth in sulfate is higher than in trichloride. A considerable increase in the degree of chloridosublimation occurs only at the temperature above 400°C.

The reaction isotherms of Bi_2O_3 with Cl_2 in the presence of SO_2 at 500–750°C resemble those of Bi_2O_3

Table 2. Distribution of bismuth between the products and solid residue at the chlorination of Bi_2O_3 and Fe_2O_3 with a mixture of Cl_2 and SO_2

t, °C	τ, min	Bi, % to initial				Fe, % to initial		
		$BiCl_3$	BiOCl	$Bi_2(SO_4)_3$	Bi_2O_3	$FeCl_3$	$Fe_2(SO_4)_3$	Fe_2O_3
300	30	4.4	62.5	19.5	13.5	19.1	18.8	62.0
	60	7.4	58.1	20.2	14.3	23.0	22.0	54.9
	90	10.3	–	22.6	–	25.2	24.7	50.0
	120	14.7	49.1	24.1	12.1	–	–	–
350	30	6.5	62.1	20.1	11.3	20.0	19.1	60.8
	60	8.6	60.3	21.2	9.9	22.0	20.9	56.9
	90	11.8	–	22.5	–	26.5	25.1	48.3
400	30	7.0	66.7	18.9	7.4	22.0	21.3	56.6
	60	10.6	62.5	19.9	7.0	23.1	21.4	55.4
	90	12.5	–	23.2	–	28.0	26.4	45.4
450	30	21.8	43.9	19.4	14.7	22.5	21.5	55.9
	60	34.4	32.4	20.5	12.7	27.3	25.0	47.6
	90	46.3	–	20.2	–	29.5	28.4	42.0

chlorination with individual chlorine: at 500–600°C in the first moment the sample mass increases due to accumulation of the intermediate reaction products. The further decrease in mass is explainable by chemical sublimation of $BiCl_3$ at the chlorination of the intermediates.

$$6BiOCl + 3SO_2 + 3Cl_2 = 4BiCl_3 + Bi_2(SO_4)_3, \quad (3)$$

$$BiOCl + SO_2 + Cl_2 = BiCl_3 + SO_3. \quad (4)$$

Comparison of rates of chloridosublimation of bismuth in the atmosphere of either Cl_2 alone or Cl_2 and SO_2 mixture shows that dilution of chlorine with sulfur dioxide accelerates chemical sublimation of $BiCl_3$ (Table 3).

Table 3. Temperature dependence of the rate of bismuth chloridosublimation

t, °C	*W*, %/min				
	Bi_2O_3			$Bi_2O_3 + Fe_2O_3$	
	Cl_2	Cl_2 + Ar	$Cl_2 + SO_2$	Cl_2	$Cl_2 + SO_2$
500	0.3	0.2	0.7	2.6	2.7
550	0.9	0.5	2.0	6.5	6.2
600	2.6	1.4	3.6	7.8	7.3
650	4.0	2.5	5.0	10.8	8.2
700	9.6	5.0	8.0	16.1	12.2
750	23.1	11.6	11.1	24.0	15.5

Noteworthy that rate of $Bi_2(SO_4)_3$ formation as a result of reactions (2) and (3) is much higher than in the atmosphere of sulfur dioxide alone. Thus, in the reaction of Bi_2O_3 with the mixed Cl_2 and SO_2 both the process, chloridosublimation of busmuth and formation of $Bi_2(SO_4)_3$, accelerate. Increase in the completeness of bismuth chloridosublimation is promoted by decrease in stability of $Bi_2(SO_4)_3$ with increase in temperature. The accelerating action of SO_2 in the region of high temperatures falls: at 750°C the rates of bismuth chloridosublimation in the mixtures of Cl_2 with either argon or sulfur dioxide are practically the same (Table 3).

Reaction of Fe_2O_3 with Cl_2 with an appreciable rate occurs only above 500°C [6], but chlorination of the most active region on the surface occurs even at 300°C (Table 1). The degree of passing $FeCl_3$ to sublimate at 300°C is less than 0.5% practically regardless the duration of the contact of oxide with chlorine. In the chlorine flow the isotherms are convex toward ordinate axis, the curves transform into one another at the change in the scale along the time axis (affine transform). This result shows that the process is limited by one and same step. The apparent activation energy of $FeCl_3$ chemical sublimation is 99 kJ mol^{-1}, as estimated from the temperature dependence of the affine transform coefficients in the temperature range 600–800°C.

Unlike bismuth oxide, reaction of Fe_2O_3 with SO_2 in the studied temperature range is restricted by adsorption and no formation of voluminous substances has been registered.

In the reaction of Fe_2O_3 with a mixture of Cl_2 and SO_2 formation of reaction products occurs even at 200°C. At 300°C and above the iron chloridosublimation proceeds with the rate higher than the rate of Fe_2O_3 chlorination in the atmosphere of Cl_2 alone (Table 1). Analysis of reaction products showed that the fractions of iron converted into chloride and sulfate are approximately equal (Table 2). The total process can be described by the following equation:

$$2Fe_2O_3 + 3SO_2 + 3Cl_2 = Fe_2Cl_6 + Fe_2(SO_4)_3. \qquad (5)$$

Thus, introduction to the system of an additional gaseous reagent (either Cl_2 or SO_2) results in appearing of new reaction pathways and increases the degree of Fe_2O_3 transformation. Like the case of Bi_2O_3, accumulation of solid phase in the temperature range 300–450°C stepwise decelerates further chlorination of Fe_2O_3, and reaction rate falls in time noticeable. At 600°C the essence of the process is changed. On the chlorination isotherm in the initial moment is seen increase in the sample mass and further it falls practically lineary. Analysis of the reaction products showed that increase in mass is defined by the formation of $Fe_2(SO_4)_3$ along the Eq. (5). Decrease in mass is a result of chemical sublimation of $FeCl_3 \cdot$ $Fe_2(SO_4)_3$ is not stable in the presence of chlorine. At the temperature above 600°C the sample mass falls monotonically. The iron chloridosublimation proceeds according to the following equartion:

$$Fe_2O_3 + 3Cl_2 + 3SO_2 = 2FeCl_3 + 3SO_3. \qquad (6)$$

The process apparent activation energy in the temperature range 600–750°C is 62.8 kJ mol^{-1}. The dependence of chloridosublimation rate on the SO_2/Cl_2 ratio in gas phase is shown in Fig. 2. The accelerating action of SO_2 falls when the temperature raises, and at 800°C sulfur dioxide behaves as inert chlorine diluting substance only.

Comparison of completeness of chemical sublimation of individual bismuth and iron chlorides showed that in the temperature range 400–700°C the rate of chloridosublimation of bismuth is much higher (Table 1).

In the mixture of Cl_2 and SO_2 the completeness of iron chloridosublimation in the temperature range 300–400°C is about those of bismuth, but at 500–700°C the amount of $BiCl_3$ passed to gas phase in a time unity is considerably higher than for $FeCl_3$.

The reaction isotherms for the Bi_2O_3–Fe_2O_3 mixture with Cl_2 (Fig. 3) are typical: at the temperature below 700°C after the induction period that is shorter at higher temperature follows practically linear decrease in the sample mass, the loss in mass is approximately 45%, and then the process sharply decelerates. We have shown earlier that the decrease in mass is a result of $BiCl_3$ sublimation while iron remains in condensed phase practically whole [11].

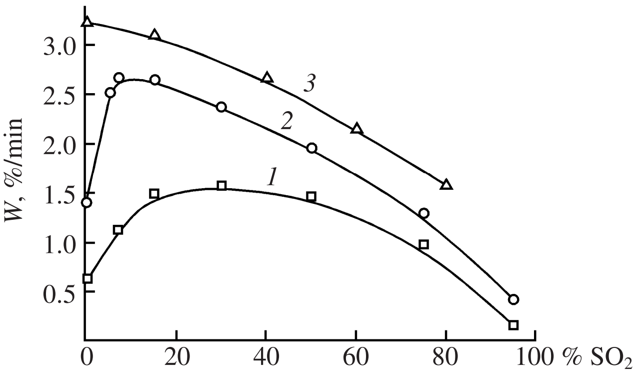

Fig. 2. Dependence of the rate of chloridosublimation on the Cl_2/SO_2 ratio in gas phase. (*1*) 700, (*2*) 750, and (*3*) 800°C.

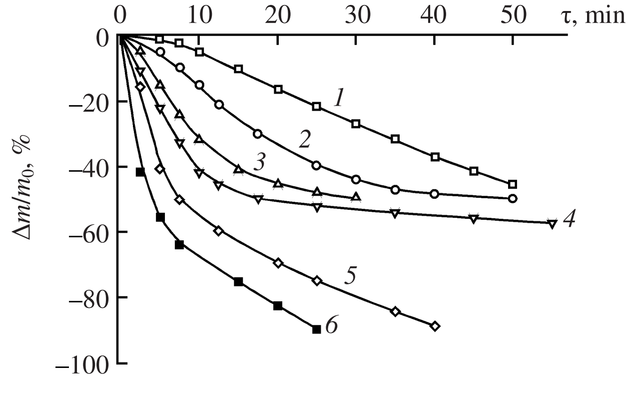

Fig. 3. Isotherms of reaction of a mixture of Bi_2O_3 and Fe_2O_3 with Cl_2. (*1*) 500, (*2*) 550, (*3*) 600, (*4*) 650, (*5*) 700, and (*6*) 750°C.

Table 4. Distribution of bismuth and iron between the product and solid residue at the chlorination of mixture of Bi_2O_3 and Fe_2O_3 with a mixture of Cl_2 with SO_2

t, °C	τ, min	Bi (% to initial)		Fe (% to initial)		
		$BiCl_3$	BiOCl	$FeCl_3$	$Fe_2(SO_4)_3$	Fe_2O_3
300	30	27.6	72.4	3.6	18.3	78.0
	60	66.7	33.3	8.5	47.7	43.7
	120	89.5	10.5	–	–	–
350	30	77.0	23.0	14.4	55.7	29.8
	60	95.9	4.1	27.0	52.4	20.5
400	30	98.7	1.3	27.2	53.2	19.6
	60	99.2	0.8	26.6	54.7	18.7
450	30	99.0	1.0	26.5	47.6	25.8
	60	99.1	0.9	28.0	49.0	23.0
500	30	99.2	–	25.6	51.8	22.6
	60	99.6	–	30.6	45.1	24.3
600	30	99.2	0.8	–	–	–
	60	100	0	31.4	47.7	20.8
650	30	99.3	0.7	–	–	–
	60	100	0	48.6	2.7	48.7

The rate of chloridosublimation of bismuth throm the mixture of oxides is much higher than in the case of individual Bi_2O_3 (Table 3). The activation energy of bismuth chloridosublimation in the presence of Fe_2O_3 is 95 kJ mol^{-1}, while for the Bi_2O_3 alone is 115 kJ mol^{-1}. Decrease in activation energy and increase in the rate of the process show that the reaction pathway is changed. This can not be a result of contact phenomena at the boards of grains because in the mechanical mixture the sufficient contact between oxides grains seems doubtful. Hence, effect of Fe_2O_3 is realized through the gas phase.

The decrease in activation energy can be defined by essential influence of iron trichloride on the chlorination process. Interaction with chlorine of the most active areas on the Fe_2O_3 surface occurs at enough low temperature (300°C). Acceleration of chemical sublimation of $BiCl_3$ in the presence of Fe_2O_3 occurs in a wide temperature range, that is the process of bismuth chloridosublimation is affected by small amount of $FeCl_3$. One can assume that iron trichloride being adsorbed on the surface of BiOCl formed intermediately at the chlorination of Bi_2O_3, promotes $BiCl_3$ formation.

$$6BiOCl + 4FeCl_3 = 6BiCl_3 + 2Fe_2O_3. \quad (7)$$

The significant difference in the rates of chemical sublimation of $BiCl_3$ and $FeCl_3$ allows to perform the process of selective bismuth chlorosublimation from Bi_2O_3 and Fe_2O_3 mixtures in the atmosphere of chlorine in the temperature range 500–700°C.

Reaction of a mixture of Bi_2O_3 and Fe_2O_3 with Cl_2 in the presence of SO_2 proceeds with a noticeable rate even at 250°C and is accompanied with the increase in the sample mass. At higher temperature after the initial increase in the mass it becomes to fall.

The results of chemical and X-ray phase analysis show that initial increase in the sample mass in the chlorination process is a result of formation in condensed phase of BiOCl and $Fe_2(SO_4)_3$ (Table 4). The further decrease in mass is a result of transfer of metal chlorides to gas phase. At 300–700°C the rate of chloridosublimation of bismuth is much higher that that of iron.

The apparent activation energy of chemical sublimation of $BiCl_3$ estimated from the degree of conversion for 15 min in the temperature range 300–400°C is 83.7 kJ mol^{-1}. The total chloridosublimation of bismuth occurs under relatively mild conditions: at 400°C at the experiment duration 60 the bismuth content in solid phase residue (as oxychloride) was 0.8%. There was no inreacted Bi_2O_3 in the residue.

Chemical sublimation of $FeCl_3$ proceeds with a noticeable rate at 300°C. But at the sublimation completeness of $BiCl_3$ below 30% (Table 4) the degree of chloridosublimation of iron is less than 4%. At removing bismuth from solid phase the completeness of iron chloridosiblimation becomes higher. The fraction of iron converted to sulfate is twice of that passed to chloride.

The data obtained show that presence of Fe_2O_3 defines sharp increase in the amount of bismuth transformed into $BiCl_3$. Therewith, in condensed phase is absent $Bi_2(SO_4)_3$ whose formation in the reaction of Bi_2O_3 with the mixture of Cl_2 and SO_2 led to deceleration of the process of bismuth chloride-sublimation. However, in the presence of Bi_2O_3 noticeable falls completeness of iron transfer into chloride but increases its fraction in the form of $Fe_2(SO_4)_3$.

The observed increase in the content of $Fe_2(SO_4)_3$ in condenced phase and increase in the rate of bismuth chloridosublimation in the temperature range 300–500°C at the reaction of Cl_2 and SO_2 with the Bi_2O_3 and Fe_2O_3 mixture can be a result of exchange reaction:

$$Bi_2(SO_4)_3 + 2FeCl_3 = 2BiCl_3 + Fe_2(SO_4)_3. \tag{8}$$

Increase in temperature leads to decrease in the role of exchange reaction, probably owing to low thermal stability of $Bi_2(SO_4)_3$. At the temperature 550–750°C unlike the region of low temperatures just after passing of gas mixture into the reactor the sample mass falls and to the vapor phase is passed not only $BiCl_3$, but also $FeCl_3$. At 600°C still occurs formation of $Fe_2(SO_4)_3$, but longer duration leads to complete sublimation of iron. This observation shows that $Fe_2(SO_4)_3$ is formed as an intermediate. At 650°C the condensed phase does not contain sulfur (Table 4), and iron chloride-sublimation proceeds mainly as the reaction (9):

$$Fe_2O_3 + 3Cl_2 + 3SO_2 = 2FeCl_3 + 3SO_3. \tag{9}$$

According to the data of Table 3, Fe_2O_3 accelerates chloridosublimation of bismuth till up to 600°C. At 650–750°C the rates of transfer of $BiCl_3$ to gas phase from individual oxide and from the Bi_2O_3 and Fe_2O_3 mixtures are similar. Increase in temperature also diminishes effect of SO_2 on the bismuth chloridosublimation. At 750°C sulfur dioxide behaves as inert diluting agent only.

Thus, on an example of chlorination of bismuth and iron oxides we showed that change in composition of gas and condensed phases leads to changes in the degree of conversion of the oxides. At the chlorination of individual oxides the completeness of chloridosublimation of bismuth and iron increases noticeable in the presence of SO_2. At higher temperatures the accelerating action of SO_2 falls. Adding of iron oxide to bismuth oxide leads to increase in the rate of bismuth chloridosublimation, therewith effect of Fe_2O_3 on the rate of $BiCl_3$ formation is much higher than that of SO_2. Moreover, at the chlorination of mixture of oxides the rate of bismuth chloridosublimation slightly falls at adding SO_2 to gas phase. At the same time, the degree of conversion of iron oxide in the presence of SO_2 grows up to 750°C. Above this temperature there is no mutual influence of chemical reactions.

EXPERIMENTAL

Roentgenograms were obtained on an ADP-2-01 diffractometer.

Reaction of Bi_2O_3, Fe_2O_3 and their mixtures with chlorine and sulfur dioxide in the temperature range 250–600°C was studied by a flow method [6]. A weighted sample 0.60–0.90 g was placed to a heated tubular reactor with an inert gas passing through it. After achieving a given temperature the inert gas flow was replaced by the flow of reacting gas. Volatile products were trapped in cooled traps. The linear gas speed was 0.02 m sec^{-1}.

The process regularities at 550–750°C were studied by gravimetric method on an installation with automatic recording of mass change in time [4]. Sensitivity of the installation is 0.001 g per one mm of its scale. The sample weight was 0.180–0.200 g. Total gas consumption was 150–200 ml min^{-1} at linear rate 0.05 m sec^{-1}. Under such conditions the rate of gas achieving the surface not limited the process. The experiments were carried out mostly at the Cl_2:SO_2 ratio 1:1.

In the work was used mechanical mixture of bismuth and iron oxides with purity grade “extra pure,” in weight ratio 1:1. For the characterization of parent samples and reaction products were applied x-ray phase analysis and commonly used procedures of chemical analysis [12].

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