

Kinetics Of The Process Of Conversion Of Production Of Calcium Chlorate With The Use Of A Filtrate Of Hydrochloric Acid Decomposition Of Washed Ck Phosphonic Concentrate

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Abstract: The process of conversion of 20-25% solutions of calcium chloride obtained by decomposition of phosphorites of Central Kizikumes with sodium chlorate at 50, 75, 100 and 125 ° C with evaporation and without evaporation of conversion solutions has been studied. The process activation energies, the order and the rate constant of the conversion reaction as well as kinetic values of the reagent consumption are determined depending on temperature, time and carrying conditions of processes. The order of the conversion process of calcium chloride with sodium chlorate equals one. It is confirmed by the fact that the conversion rate constant calculated on the basis of experimental data remains practically constant for each temperature. Also, the linear dependence of $\lg(C_0 - C_\tau)$ on τ also indicates the first order of the conversion process of calcium chloride with sodium chlorate. The rate constant of conversion increases with a rise in temperature. Its dependence on temperature obeys Arrhenius law. To establish values of the conversion rate constant for different temperatures, the constants (K_0) for 20 and 25% solutions of calcium chloride have been calculated by Arrhenius equation and the dependence equation of $\lg K$ on $1/T$ has been derived. It is confirmed by the linear graphical dependence of $\lg K$ on $1/T$. The expediency of carrying out the process of conversion with evaporation at a temperature of 100 ° C is established.

Based on the polythermal and isothermal diagrams of the solubility of the system $\text{CaCl}_2\text{-NaClO}_3\text{-H}_2\text{O}$ [1] the possibility of obtaining a solution of calcium chlorate by interaction in an aqueous medium of sodium chlorate and calcium chloride, with the release of sodium chloride into the solid phase is shown. The work [2] presents data on the study of direct precipitation of hydrochloric acid extract obtained by decomposition of the mineralized mass (14,60 % P_2O_5) and phosphate rock (17,09 % P_2O_5) with hydrochloric acid, a fertilizer precipitate of the composition 23,21-23,69 % P_2O_5 и 25,07-27,22 % P_2O_5 , respectively. The

main limiting factor in the implementation of industrial production of fertilizer precipitate by the hydrochloric acid method is the utilization of calcium chloride formed during processing. Therefore, in order to utilize solutions of calcium chloride by processing calcium defoliant into chlorate, the process of conversion of solutions of calcium chloride with sodium chlorate was studied depending on the temperature and duration of the process.

The study was carried out at temperatures 50, 75, 100, 125°C and the duration of the experiments 30, 60, 90, 120 minutes with and without evaporation of conversion solutions. In a round bottom flask with a capacity 500 cm³, equipped with a stirrer, loaded with 100 g of 20.0 - and 25.0% aqueous solutions of calcium chloride and an equivalent amount of sodium chlorate. The flask was placed in a thermostat with a predetermined temperature and vigorously stirred. After the required time interval, the liquid phase was separated from the sediment and the appropriate chemical analysis was carried out. The content of chlorate and chlorine ions was determined by volumetric permanganometric and argentometric [3,4], the amount of sodium and calcium, respectively, by flame photometry [5] and complexometric [6,7] methods of analysis.

As noted in [2], the analysis of the chemical composition of the filtrate obtained in the filtration process showed that at a 45% acid rate, the calcium chloride content was 18.88%, with an increase in the acid rate from 55 to 75%, the calcium chloride content increased 1.06 -1.16 times, respectively. At an acid rate of 75%, hydrochloric acid pulp has a high content of calcium chloride (table 1.).

Table 1. Chemical composition of the filtrate obtained by filtration of hydrochloric acid pulp, %

Acid rate	CaO	Cl ⁻	CaCl ₂	H ₂ O
1-filtrate				
45	9,52	12,07	18,88	81,12
55	10,11	12,81	20,03	79,97
65	10,59	13,43	20,99	79,01
75	11,04	13,99	21,88	78,12
2-filtrate				
45	2,37	3,00	4,69	95,31

55	2,33	2,96	4,62	95,38
65	2,43	3,08	4,81	95,19
75	2,49	3,15	4,93	95,07

In order to issue practical recommendations, the process of conversion of calcium chloride with sodium chlorate was investigated depending on temperature and time duration. The study was carried out at temperatures 50, 75, 100, 125°C and the duration of the experiments 30, 60, 90, 120 minutes, with and without evaporation of conversion solutions. A round-bottom flask with a capacity of 500 cm³ equipped with a stirrer was loaded with 200 g 20,0 and 25,0% aqueous solutions of calcium chloride and an equivalent amount of sodium chlorate. The flask was placed in a thermostat with a predetermined temperature and vigorously stirred. After the required time interval, the liquid phase was separated from the sediment and the appropriate chemical analysis was carried out. The content of chlorate and chlorine - ions was determined by volumetric permanganometric and argentometric methods, the amount of sodium and calcium, respectively, by flame photometry and complexometric [191, 192; 9-12s., 193; 56s., 194]. On the basis of the data obtained, the degree of conversion of calcium chloride with sodium chlorate and the consumption of the starting components during conversion were established (Table 2.9-2.10, Fig. 2.8-2.9).

From Table 2.9 and Figure 2.8 it can be seen that the degree of conversion depends significantly on temperature and increases with its growth. For processes of 20 and 25% solutions for 60 minutes without removing water, the degree of conversion at 323, 348, 373, and 398 ° K, respectively, is 27.3; 32.7; 34.6 and 44.1%; 33.2; 38.2; 43.6 and 50.2%. After 90 minutes, the conversion reaches 27.8; 33.9; 39.8; 45.9% & 33.7; 38.9; 43.9; 50.9%, respectively, for 20 and 25% calcium chloride solutions, at temperatures of 323; 348, 373 and 398°K.

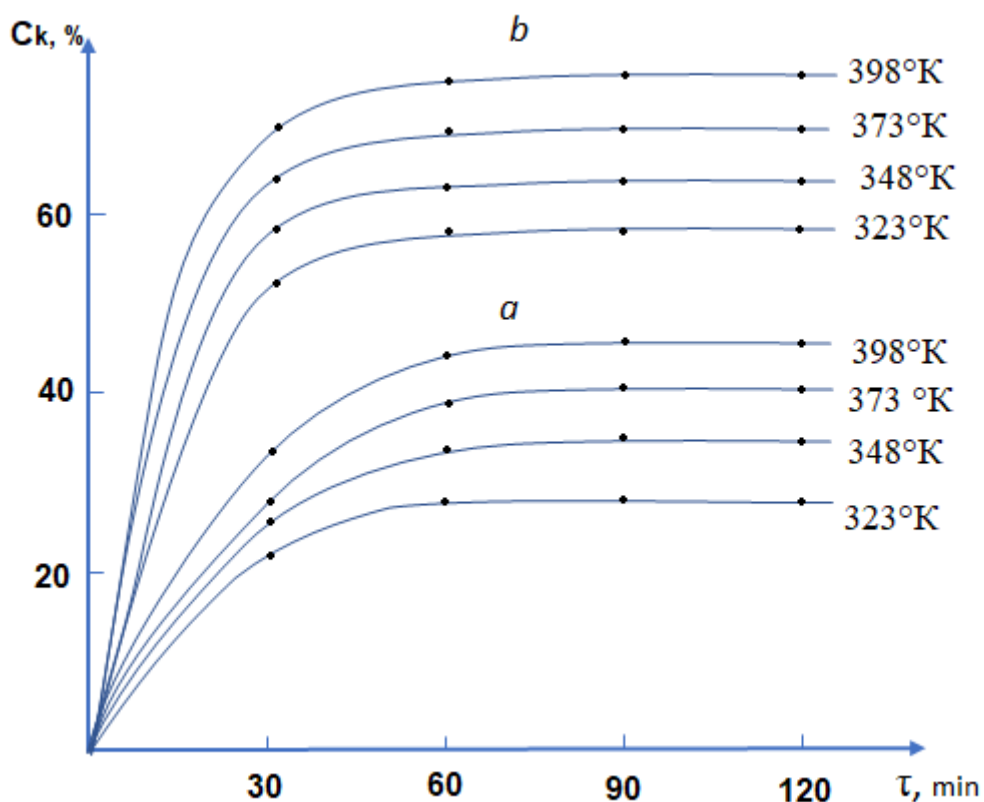


Figure 2.8. Dependence of the degree of conversion of calcium chloride with sodium chlorate on the temperature and duration of the process without evaporation (a), and with evaporation (b) of conversion solutions

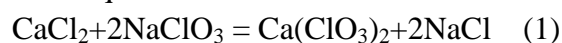
In the case of a conversion process with evaporation, the data have higher values: for 323; 348, 373 и 398°K after 60 minutes the conversion rate is 57,0; 63,4; 69,2; 75,0% и 59,0; 63,0; 68,6; 74,2% for 20- and 25% calcium chloride solutions, respectively. The subsequent increase in the duration of the conversion practically does not lead to an increase in the degree of conversion of calcium chloride with sodium chlorate. When 373°K and the duration of the experiment 120 minutes, the degree of conversion was 70.0 and 70.8%, respectively. At this temperature, an increase in the duration of the experiment from 90 to 120 minutes leads to an increase in the degree of conversion by only 0.2 and 1.0%.

Table 2.9 Dependence of the rate constant and the degree of conversion on the temperature and duration of the process when converting calcium chloride solutions without evaporation

Temperature, °K	Time, (τ), min.	Content Ca(ClO ₃) ₂ in liquid phase, %	Conversion rate, (C _κ)%	Rate constant, K·10 ⁻² , τ ⁻¹	lgK	lg(C ₀ - C _τ)	Activation energy, (E), kJ / mol
For 20% calcium chloride solution							
323	30	8.52	22.4	0.008623	-2.0643	1.4591	9,108·10 ³
	60	11.10	27.3	0.005958	-2.2248	1.4183	
	90	11.69	27.8	0.004183	-2.3785	1.4084	
	120	12.13	28.9	0.003268	-2.4857	1.4008	
	The average			0.005508	-2.2883	1.4216	
348	30	10.5	25.0	0.011022	-1.9577	1.4281	9,108·10 ³
	60	14.9	32.7	0.008501	-2.0705	1.3503	
	90	15.5	33.9	0.005969	-2.22409	1.3385	
	120	16.14	35.0	0.004708	-2.3271	1.3255	
	The average			0.007551	-2.1448	1.3606	
373	30	11.7	28.0	0.012549	-1.9014	1.4082	9,108·10 ³
	60	17.4	34.6	0.010472	-1.9799	1.2988	
	90	17.8	39.8	0.007208	-2.1422	1.2900	
	120	18.5	40.9	0.005709	-2.2436	1.2742	
	The average			0.008984	-2.0668	1.3178	
398	30	13.5	32.5	0.014982	-1.8245	1.3766	9,108·10 ³
	60	19.7	44.1	0.012519	-1.9024	1.2455	
	90	20.8	45.9	0.009065	-2.0426	1.2175	
	120	20.9	46.2	0.006848	-2.1644	1.2148	
	The average			0.0086072	1.9835	1.5136	
Table continuation 2.9							
For 25% calcium chloride solution							
323	30	14,2	30,5	0,01212	-1,91657	1,5105	7,252·10 ³
	60	15,5	33,2	0,006741	-2,1713	1,4927	
	90	15,7	33,7	0,004566	-2,3405	1,4899	
	120	15,9	34,0	0,003478	-2,4587	1,4871	
	The average			0,006725	-2,2217	1,4951	
348	30	15,9	34,0	0,013914	-1,8565	1,4871	7,252·10 ³
	60	17,8	38,2	0,008022	-2,0972	1,4594	
	90	18,1	38,9	0,005460	-2,2628	1,4548	
	120	18,6	39,8	0,004245	-2,3721	1,4472	
	The average			0,007910	-2,1472	1,4621	
373	30	17,2	37,0	0,015356	-1,8137	1,4683	7,252·10 ³
	60	20,3	43,6	0,009534	-2,0207	1,4199	
	90	20,5	43,9	0,006442	-2,1909	1,4166	

	120	20,5	44,0	0,004831	-2,3159	1,4166	
	The average			0,009041	-2,0853	1,4303	
398	30	19,2	41,3	0,017712	-1,7518	1,4377	$7,252 \cdot 10^3$
	60	23,4	50,2	0,011625	-1,9346	1,3655	
	90	23,7	50,9	0,007895	-2,1027	1,3598	
	120	23,9	51,4	0,005994	-2,2223	1,3560	
	The average			0,010806	-2,0028	1,4548	

Due to the fact that during the first 60-90 minutes of conversion, such an amount of sodium chloride is formed by reaction (1), which further negatively affects the course of this reaction in an aqueous medium.



Analysis of the kinetic curves of the consumption of calcium chloride and sodium chlorate per 100 g of a 30.0% solution of calcium chloride during conversion at 323-393 ° K indicates that the amount of consumed starting components increases in the first 60-90 minutes, and after 90 minutes practically does not change. Temperature rise from 323 to 398°K leads to an increase in the consumption of calcium chloride and sodium chlorate during conversion.

The order of the reaction of the conversion process was determined using the kinetic equation of the first order:

$$K = 2,303/\tau \cdot \lg C_0 / (C_0 - C_\tau) \quad (2) \quad [8-10]$$

where C_0 and C_τ – the concentration of calcium chloride (or sodium chlorate), respectively, at the initial stage of conversion and over the elapsed period of time (τ), K - conversion rate constant.

According to the data obtained, the order of the reaction of the conversion of calcium chloride with sodium chlorate is equal to unity. This is confirmed by the fact that the conversion rate constant calculated by Eq. (2) based on experimental data remains practically constant for each temperature (Table 2.10). Moreover, the straightforward dependence $\lg(C_0 - C_\tau)$ from τ also indicates the first order of the conversion of calcium chloride with sodium chlorate. The conversion rate constant increases with increasing temperature (Table 2.11). Its dependence on temperature obeys Arrhenius's law and is also confirmed by a straight-line graphical dependence $\lg K$ on $1/T$.

When carrying out conversion with evaporation, the intensity of the process is significantly accelerated, as evidenced by the data presented in tables 2.10 and 2.12.

Table 2.10. Dependence of the rate constant and the degree of conversion on the temperature and duration of the process when converting 30 and 35% calcium chloride solutions with evaporation

Temperature, °K	Time, (τ), min.	Content Ca(ClO ₃) ₂ in liquid phase, %	Conversion rate, (C _K)%	Rate constant, K·10 ⁻² , τ ⁻¹	lgK	lg(C ₀ - C _τ)	Activation energy, (E), kJ / mol
For 20% calcium chloride solution							
323	30	24,2	52,0	0,03488	-1,4574	1,1173	7,063·10 ³
	60	26,5	57,0	0,02066	-1,6848	1,0334	
	90	26,9	58,0	0,01419	-1,8480	1,0171	
	120	27,6	59,1	0,01122	-1,9499	0,9868	
	The average			0.0202375	-1.7350	1.03865	
348	30	27,5	59,0	0,04389	-1,3576	0,9912	7,063·10 ³
	60	29,5	63,4	0,02608	-1,5836	0,8912	
	90	29,7	63,8	0,01768	-1,7525	0,8808	
	120	29,9	64,2	0,01348	-1,8703	0,8692	
	The average			0.0252825	-1.6410	0.9081	
373	30	29,8	64,0	0,05351	-1,2716	0,8751	7,063·10 ³
	60	32,2	69,2	0,03316	-1,4794	0,7076	
	90	32,5	69,8	0,02278	-1,6424	0,6812	
	120	32,7	70,0	0,017443	-1,7584	0,6628	
	The average			0.03172325	-1.53795	0.731675	
398	30	32,2	69,0	0,06634	-1,1782	0,7076	7,063·10 ³
	60	34,9	75,0	0,04573	-1,3397	0,3802	
	90	35,3	75,7	0,032516	-1,4879	0,3011	
	120	35,4	76,0	0,024812	-1,6053	0,2787	
	The average			0.0423495	1.402775	0.4169	
Table continuation 2.10							
For 25% calcium chloride solution							
323	30	25,3	54,4	0,02610	-1,5836	1,3284	7,299·10 ³
	60	27,5	59,0	0,01487	-1,8277	1,2810	
	90	27,9	59,9	0,01015	-1,9935	1,2718	
	120	27,9	60,0	0,00761	-2,1186	1,2718	
	The average			0.0146825	-1.88085	1.28825	
348	30	27,9	60,0	0,030443	-1,5165	1,2718	7,299·10 ³
	60	29,4	63,0	0,016613	-1,7796	1,2355	
	90	29,8	63,8	0,011338	-1,9455	1,2253	
	120	29,8	64,0	0,0085	-2,0706	1,2253	
	The average			0.016724	-1.82805	1.239475	
373	30	29,5	63,3	0,03343	-1,4758	1,2329	7,299·10 ³
	60	31,9	68,6	0,04198	-1,3769	1,1673	

	90	32,1	69,0	0,01297	-1,8871	1,1613	
	120	32,9	70,8	0,010202	-1,9913	1,1367	
	The average			0.0246455	1.682775	1.17455	
398	30	32,2	69,1	0,03915	-1,4073	1,1584	$7,299 \cdot 10^3$
	60	34,6	74,2	0,02261	-1,6457	1,0792	
	90	34,8	74,7	0,01526	-1,8164	1,0719	
	120	36,8	79,0	0,01299	-1,8864	0,9912	
	The average			0.0225025	1.68895	1.075175	

At 75 ° C after 60 minutes, the removal of 15.0-18.5% of water from the reaction mixture leads to an increase in the degree of conversion to 65.0-66.0% for 20 and 25% calcium chloride solutions. As the temperature rises, the conversion process and the degree of water removal accelerate. With a process duration of 60 minutes at 100 ° C, the degree of conversion for 20 and 25% solutions was 70.0 and 70.8%, respectively. At 125 ° C, the conversion rate reaches 76.0-79.0%.

In order to establish the values of the conversion rate constant for different temperatures, the constants were calculated (K_0) for 20 and 25% calcium chloride solutions by the Arrhenius equation.

$$K = K_0 e^{E/RT} \quad (3)$$

and the equation of dependence is derived $\lg K$ on $1/T$.

Let's convert more complex functions to linear ones. After taking the logarithm of equations (3), we obtain:

$$\lg K = \lg K_0 - E/2,303 \cdot 1,987 \cdot 1/T \quad (4)$$

In order to shorten the notation, we introduce new designations

$$\lg K = \eta; \lg K_0 = a; b = E/2,303 \cdot 1,987 = E/4,184; 1/T = \xi$$

$$\text{We get: } \eta = a - b \cdot \xi \quad (5)$$

Making up the relationship:

$$b_{2,1} = \eta_2 - \eta_1 / \xi_1 - \xi_2; b_{3,2} = \eta_3 - \eta_2 / \xi_2 - \xi_3; b_{3,4} = \eta_3 - \eta_1 / \xi_1 - \xi_3; b_{4,1} = \eta_4 - \eta_1 / \xi_1 - \xi_4$$

and calculating the individual values "b" based on the experimental data, we find the average value "b".

The calculation of the average value "a" is found by the formula:

$$a = \Sigma \eta + b \cdot \Sigma \xi / 4 \quad (6).$$

For a process using 30% calcium chloride with evaporation, the data have the following values. Substituting the calculated values "a" and "b" into equation (5) we will have:

$$\eta = -6,0527133 - 373,965 \cdot \xi \quad (7);$$

$$\lg K = -6,0527133 - 373,965 \cdot (1/T) \quad (8).$$

The value of the apparent activation energy (E), calculated by the formula $E = b \cdot 4,576$, was 1711,264 kJ / mol or $7,16335 \cdot 10^3$ kJ / mol. Substituting the calculated value "a" into $\ln \lg K_0 = a$ get:

$$\lg K_0 = -6,0527157.$$

$$\text{From here } K_0 = 8,857 e^{-7} \text{ or } K_0 = 0,8857 \cdot 10^{-7}.$$

After substitution of values K_0 and E the empirical Arrhenius equation (3) takes the form

$$K = 0,8857 \cdot 10^{-6} \cdot \exp(1711,26/T) \quad (9).$$

When carrying out conversion with evaporation after calculations, the following equation is derived:

$$\lg K = -6,0527157 - 373,965 \cdot \frac{1}{T} \quad (10).$$

For the case using a 35% calcium chloride solution, the data have the following meanings. Substituting the calculated values "a" and "b" into equation (5) we will have:

$$\eta = -5,968031 - 386,31 \cdot \xi \quad (7);$$

$$\lg K = -5,968031 - 386,31 \cdot 1/T \quad (8).$$

The value of the apparent activation energy (E) [20, 66-73], calculated by the formula $E = b \cdot 4.576$, was 1767.75 cal / mol or $7,39982 \cdot 10^3$ kJ / mol. Substituting the calculated value "a" in $\lg K_0 = a$:

$$\lg K_0 = -5,968031.$$

$$\text{From here } K_0 = 1,0764 \cdot 10^{-6}.$$

After substitution of the values of K_0 and E, the empirical Arrhenius equation (3) takes the form

$$K = 1,0764 \cdot 10^{-6} \cdot \exp(1767,75/T) \quad (11).$$

Thus, from the results of the studies carried out, it follows that to obtain a defoliant with an optimal content of the active substance, it is recommended to carry out the conversion of 20-25% solutions of calcium chloride with sodium chlorate at a molar ratio of the components 1:2 for 60 minutes at 373°K with evaporation, as a result of which 31.9-32.2% solutions of calcium chlorate defoliant can be obtained.

Data on the change in the content of chlorate and calcium chloride in the liquid phase depending on the duration of the conversion at 373 ° K are presented in Figure 2.11. According to the data obtained, the content of calcium chlorate in the hot solution after 60 minutes of the duration of the conversion reaches 31.9%, and calcium chloride decreases from 20.0-25.0 to 6.4-7.0%.

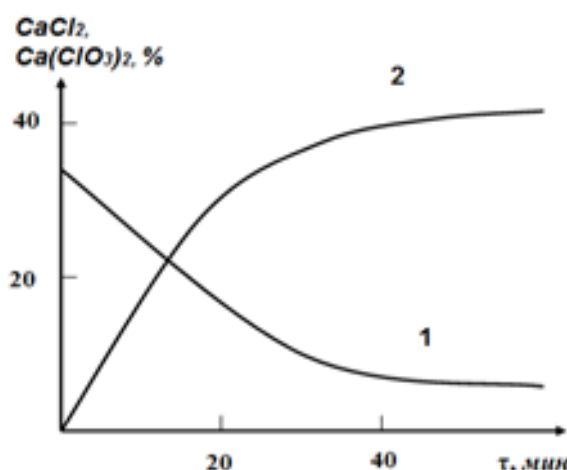


Figure 2.11. The dependence of the change in the content of chloride (1) and chlorate (2) calcium in the liquid phase on the duration of conversion at 373 ° K

We present the calculations for obtaining liquid calcium chlorate defoliant by converting

sodium chlorate with 20.0-25.0% aqueous solution of calcium chloride.

Figure 2.12 presents a diagram to justify the conversion of calcium chloride with sodium chlorate. When mixing a solution of calcium chloride with an equimolar amount of sodium chlorate at 373 ° K, a pulp is formed, the composition of which is determined by the point «P₁₀₀». Pulp coordinates «P₁₀₀» according to the Eneke index in ion - eq. for 1 mol of salts correspond Ca^{2+} -0,4999, 2Na^+ -0,5001, 2Cl^- - 0,4999, 2ClO_3^- - 0,5001, H_2O -5,5632.

The general equation of the conversion process is expressed by the equality:

1ME «P₁₀₀»

Ca^{2+} -0,4999

2Na^+ -0,5001

$x\text{CaCl}_2 + y\text{NaClO}_3 + z\text{H}_2\text{O} = 2\text{Cl}^- - 0,4999$

$2\text{ClO}_3^- - 0,5001$

$\text{H}_2\text{O} - 5,5632$

From here $x=0,4999$, $y=0,5001$, $z=5,5632$

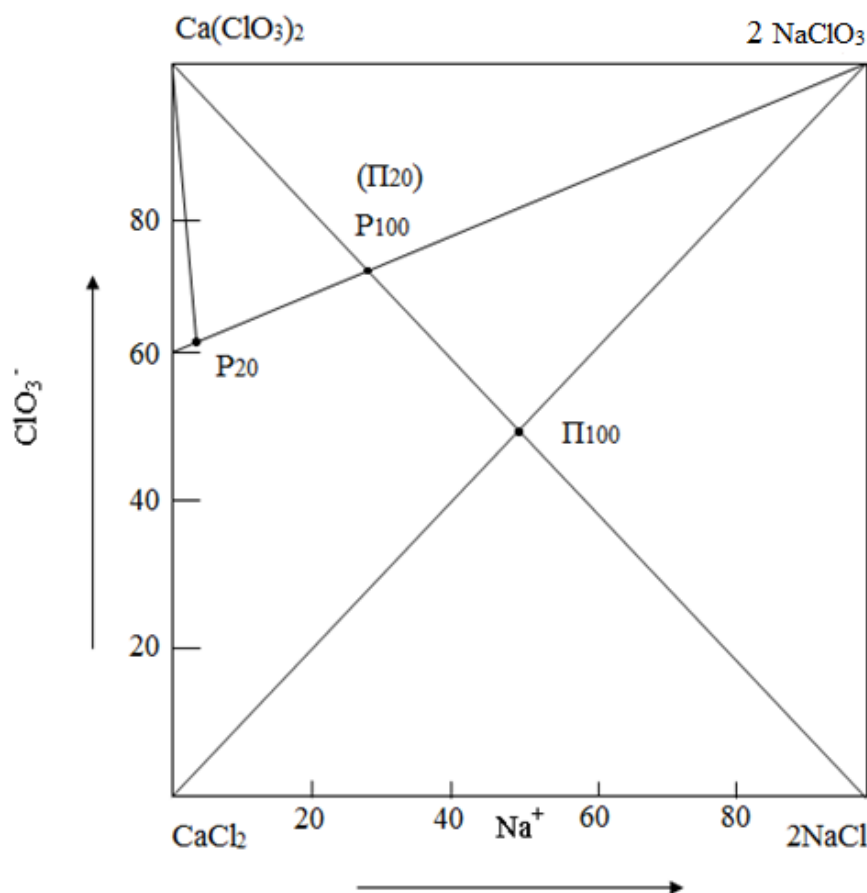
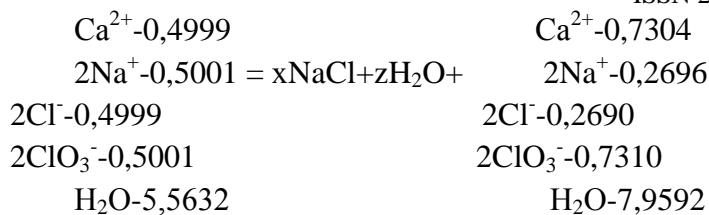


Figure 2.12. Diagram to justify the process of obtaining liquid chlorate calcium defoliant

This means that when mixing 0.4999 mol of calcium chloride and 0.5001 mol of sodium chlorate and 5.5632 mol of water, 1 IU (molar unit) pulp is formed «P₁₀₀» with sodium chloride precipitation. After separation of the pulp «P₁₀₀» into the solid and liquid phases, we obtain sodium chloride and a solution, the coordinates of which in Fig. 2.12 correspond to the point «P₁₀₀». During filtration of the hot slurry, partial evaporation of water also occurred.

1 ME «II₁₀₀»

a ME «P₁₀₀»



We compose partial equations for cations, anions and water

$$\text{Ca}^{2+}-0,4999 = a \cdot 0,7304$$

$$2\text{Na}^{+}-0,5001 = x+a \cdot 0,2696$$

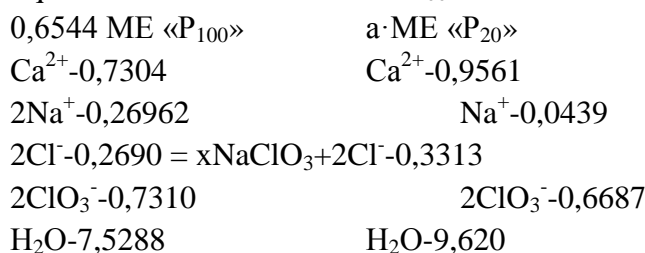
$$2\text{Cl}^{-}-0,4999 = x+a \cdot 0,2690$$

$$2\text{ClO}_3^{-}-0,5001 = a \cdot 0,7310$$

$$\text{H}_2\text{O}-4,4611 = z+a \cdot 7,9592$$

After solving the partial equations, we find, $a = 0.6844$, $x = 0.3156$, $z = 0.1159$. Thus, with filtration of 1 IU of pulp «P₁₀₀» 0.3156 mol of sodium chloride is released, 0.1159 mol of water evaporates and a 0.6844 IU solution is formed «P₁₀₀». After cooling to 293 ° K solution «P₁₀₀» with a temperature of 373 ° K, 0.6844 IU pulp is formed «P₂₀» with sodium chlorate precipitate. The coordinates of this pulp correspond to the coordinates of the solution «P₁₀₀». The final composition of the newly formed solution is determined by the continuation of the line $2\text{NaClO}_3 - \text{P}_{100}$ at the point «P₂₀» (fig.2.12). Solution coordinates «P₂₀» in ion - equivalents 1 mol of salts corresponds to $\text{Ca}^{2+}-0,9561$, $2\text{Na}^{+}-0,0439$, $2\text{Cl}^{-} - 0,3313$, $2\text{ClO}_3^{-} - 0,6687$, $\text{H}_2\text{O}-9,620$.

Cooling process equation 0.6544 IU solution «P₁₀₀» from 373°K before 293°K.



After drawing up and solving partial equations for cations, anions and water, we find, $a=0,5228$, $x=0,1616$. So when cooled, 0.6844 IU of solution «P₁₀₀» before 293°K formed 0.6844 IU pulp «P₂₀», which, when filtered, is divided into 0.1616 mol of sodium chlorate and 0.5228 IU of solution «P₂₀», containing 40,24% calcium chlorate, 9.22% calcium chloride, 0.742% sodium chloride and 50.04% water. The resulting solution «P₂₀» is a clear solution with a slightly yellowish tint and is a ready-made liquid chlorate calcium defoliant. Thus, the process of obtaining liquid calcium chlorate by the conversion of calcium chloride with sodium chlorate is carried out according to the scheme $\text{P}_{100} - \text{P}_{100}(\text{P}_{20}) - \text{P}_{20}$, shown in Fig.2.12.

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