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Izvestiya Vysshikh Uchebnykh Zavedeniy SSSR
"Khimiya i Khimicheskaya Tekhnologiya" No. 4, 533-538 (1965)

Translated from the Russian

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The thermal decomposition of perchlorate of ammonium (PA), pure as well as with additives, is dealt with in many papers. The kinetics of the decomposition have been studied essentially by measuring the pressure or volume of the evolving gases.

Bircumshaw and Newman[1] investigated the decomposition of PA at 215°to 275°C. They found that the decomposition of PA starts on the surface and propagates in the form of half-spheres which later combine into a continuous front. The process ceases after the decomposition of about 30 percent of the salt. The residue represents a friable powder which is identical with the original PA. The energy of activation of the decomposition of PA is $E = 27.8$ kcal/mole (1 kcal = 4.1868 joules) for the orthorhombic and $E = 18.9$ kcal/mole for the cubic form.

Galwey and Jacobs[2] investigated the decomposition of PA by the manometric method and found $E = 24.6$ kcal/mole for the orthorhombic and cubic forms of the PA. They confirmed the indication[1] that at a temperature above 300°C the PA decomposes completely. Galwey and Jacobs[3] found $E = 17.5$ kcal/mole for the decomposition of PA and $E = 30$ kcal/mole for the decomposition of the residue from the low-temperature reaction at a temperature above 350°C. While measuring the delay in the ignition as a function of the temperature, they found $E = 41.1$ kcal/mole[4].

Rayevskiy and Manelis[5], while measuring by the method of microphotography the rate of growth of the nuclei of the decomposition of the PA, found $E = 31$ to 33 kcal/mole. Sun Tsuan-tsai[6] studied, by the manometric method, the decomposition of PA and found by different methods of calculation that the energy of activation was about 30 kcal/mole. According to data by Osada and Sakamoto[7], the energy of activation of the decomposition of PA depends on the size of the particles. The energy of activation of the decomposition of PA with additives of metal oxides is, according to literature data, about 30 kcal/mole and depends little on the chemical nature of the oxide. A study was made of the decomposition of PA with additives of the oxides MnO_2[8,9,10], Cu_2O[11,12,10], CuO[13,10], ZnO[14,10], Fe_2O[15], MgO[16,9], Cr_2O_3[9,10], Ni_2O_3, $Co_2O_3+Co_3O_4$[9], as well as the chlorides of metals[7]. The action of the catalytic additives is explained by their participation in the process of electron transfer from ClO_4^- to NH_4^+.

The purpose of this work was to study the thermal decomposition of PA, pure and in the presence of oxides, chlorides, carbonates, and oxalates of certain metals.

EXPERIMENTAL SECTION

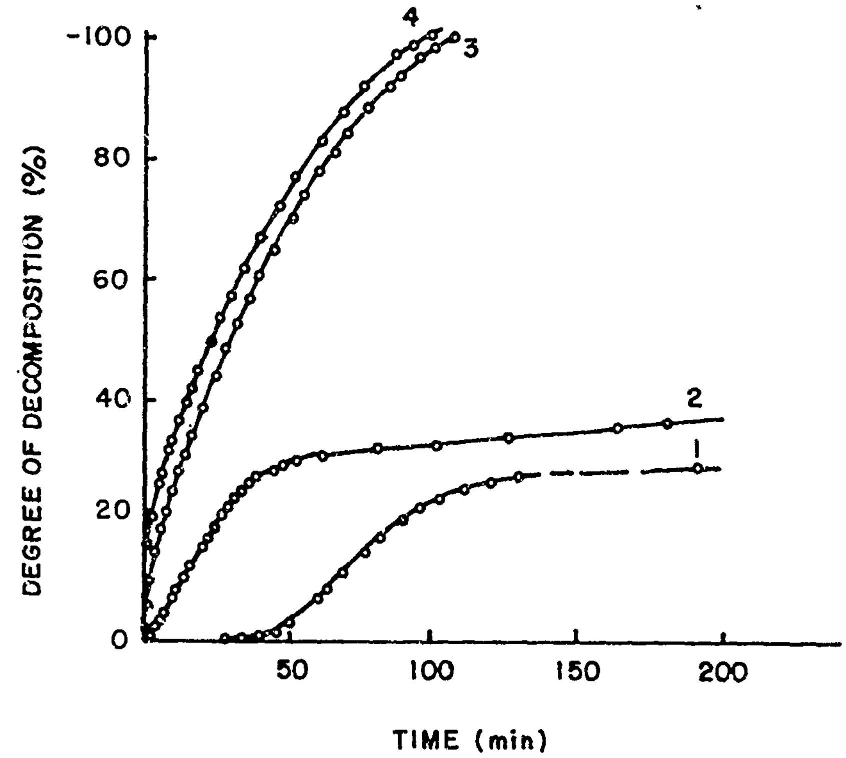
The study of the kinetics of thermal decomposition was conducted by the gravimetric method. The decomposition was conducted in an air atmosphere at atmospheric pressure. The free removal of the decomposition products excluded their influence on the decomposition of PA[6]. The substance (about 150 mg) was placed in a glass cup which was, by means of a glass capillary, lowered into an oven previously heated to the given temperature and suspended on a lever of a torsion balance VT-200 (accuracy of weighing 0.2 mg). The temperature in the oven was controlled with an accuracy of ±1°C.

The PA, recrystallized from water, was dried at 100°to 110°C, ground, and sifted through a 018 sieve. In all the experiments, use was made of one specimen of PA which was stored in a desiccator over anhydrous calcium chloride.

The kinetics of decomposition of pure PA were studied within the temperature limits of 214° to 470°C. Figure 1 shows the decomposition curves α - t (degree of decomposition--time) of pure PA and the decomposition curve at 410°C of the residue from the low-temperature reaction (at 230°C). Decomposition of the orthorhombic form of PA ($t <$ 240°C) is characterized by the existence of an induction period, period of acceleration, and period of drop in the speed of decomposition which corresponds to the formation, growth, and intergrowth of the centers of decomposition into a continuous front[1]. The decomposition stops upon the achievement of $\alpha = 30$ percent, after which, a white, porous, slightly sintered residue of PA remains. The maximum rate of decomposition develops for α of about 10 percent.

The decomposition of the cubic form of PA (240° to 300°C) proceeds without an induction period. The rate of decomposition reaches a maximum for $\alpha = 3$ to 5 percent. On the decomposition curve, one can distinguish three periods: period of acceleration, period of rapid decomposition, and period with a considerably smaller, almost constant rate. The changeover to gradual decomposition takes place for $\alpha = 20$ to 30 percent; besides, the higher the temperature of decomposition, the greater the α at which this changeover takes place.

At 300° to 380°C, the reproducibility of the kinetic curves is poor as a result of a change in the mechanism of the decomposition of the PA[3]. At a temperature above 380°C, the rate of decomposition of PA decreases with time. The decomposition is complete.



1-230°C, 2-281°C, 3-410°C, and 4-410°C (residue from low-temperature reaction).

Figure 1. Decomposition Curves of Ammonium Perchlorate

A study was made of the decomposition of the residue from the low-temperature reaction (at 230° C). For this purpose, the residue, after the reaction had ceased, was transferred to another oven and its decomposition was studied. The decomposition curves of the residue are reproducible at temperatures above 380° C. Decomposition was complete. The maximum rate of decomposition of the residue was greater for the original PA. The residue from the low-temperature reaction burns up 1 to 2 minutes after immersion of the specimen in the oven which had been heated to 450° C, whereas the original PA catches fire at 470° C. At a temperature above 300° C, a noticeable sublimation of the PA was observed.

A kinetic analysis of the decomposition curves was conducted with the help of the Yerofeyev equation[17],

$$-\ln(1-\alpha) = kt^{n}, \tag{1}$$

where α is the degree of decomposition at the time t, and k is a constant. The values of k and n were determined from the curves of $\lg[-\lg(1-\alpha)]$ - $\lg t$. The values of the constant, k, obtained from these curves, were recalculated in accordance with the Sakovich equation[17],

$$K = nk.^{/n}, \tag{2}$$

where K is a rate constant with the dimension of the rate constant of the monomolecular reaction (t^{-1}), and k is the constant from the Yerofeyev equation.

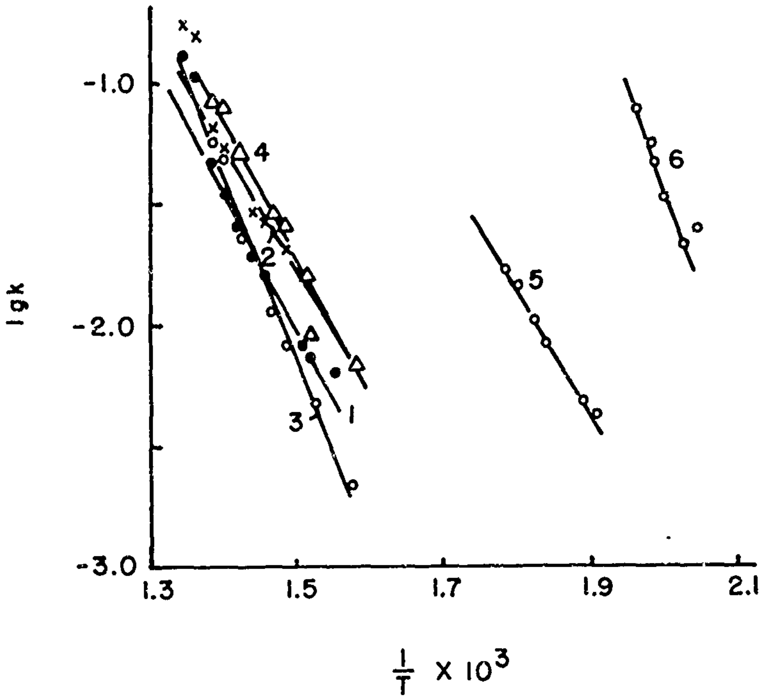
The limits of applicability of the Yerofeyev equation are listed in Table I. The straight lines, $\lg[-\lg(1 - \alpha)]-\lg t$, corresponding to the decomposition of the PA and the residue from the low-temperature reaction, at a temperature above 380°C have breaks at $\alpha = 40$ to 50 percent. At the same time, different values of n and k are obtained for the start and end of the decomposition.

Table I. Kinetic Characteristics of the Thermal Decomposition of Ammonium Perchlorate

Decomposition Temperature, °C	Limits of Applicability of the Yerofeyev Equation of α, %	Index of Degree, n	Preexponential Multiplier, C	Energy of Activation, E (kcal/mole)
214 to 236	0 to 12	4.5	$1.6 \cdot 10^{16}$	40.1
250 to 300	0 to 20	1.2	$1.3 \cdot 10^{8}$	25.1
400 to 470	10 to 50	0.8	$2.0 \cdot 10^{7}$	28.3
400 to 470	50 to 90	1.1	$1.1 \cdot 10^{6}$	23.7
330 to 450*	0 to 40	0.6	$4.5 \cdot 10^{10}$	39.1
330 to 450*	40 to 80	1.0	$5.9 \cdot 10^{9}$	35.5

*Residue from low-temperature reaction.

The magnitudes of the energy of activation were determined from the curves of $\lg K = 1/T$ (Figure 2). It is seen in Figure 2 that the phase transition at 240°C and the change in the decomposition mechanism at 300° to 380°C[3] cause a sharp drop in the rate constant. There is just as sharp a change at these temperatures in the value of the maximum rate of decomposition of PA. The resulting value of $n = 4.5$ for the orthorhombic form corresponds to the value by Galwey and Jacobs[4]. During decomposition of the cubic form of PA, the index of the degree is close to unity and even less than it; i.e., the reaction becomes monomolecular and changes into the diffusion realm[17].



1-t > 380°C, α< 50 Percent; 2-t > 380°C, α>50 Percent; 3-Residue from Low-Temperature Reaction t > 380°C, α<40 Percent; 4-Residue from Low-Temperature Reaction, t > 380°C, α = 40 - 80 Percent; 5-t = 240°- 300°C; 6-t = 214° - 236°C.

Figure 2. Dependence of lgK on 1/T During Decomposition of Ammonium Perchlorate

The value of the energy of activation which we obtained for the orthorhombic form of PA was somewhat higher than in other words[1, 2,3] this can be explained by the fact that, during decomposition of PA in a closed space, side reactions can take place which influence the decomposition. In our experiments, the removal of the decomposition products was free. The value of the energy of activation for the decomposition of the residue from the low-temperature reaction is at t > 380°C close to E obtained during[4] a study of the delay time of ignition. The decomposition of the original PA at this temperature proceeds with a considerably smaller E, which is possibly connected with the influence of the intercrystallite material.

THERMAL DECOMPOSITION OF PA WITH ADDITIVES

As additives in the thermal decomposition of PA, use was made essentially of compounds (oxides, chlorides, carbonates, and oxalates) of d-elements of the IV period of the Mendeleyev periodic system (from V to

Zn) in the form of standard ("analytical grades" or "pure" grades) or of preparations synthesized by us[18,19,20]. The additives were ground and sifted through a 018 sieve. The additive content was most frequently 5 percent by weight: such a large amount of additive was taken in order to determine clearly the nature of its influence. The mixtures were prepared by mechanical mixing of the additive samples and of the PA (the same specimen of PA was used as during the decomposition of the pure PA). The ready mixtures were stored in a desiccator over anhydrous calcium chloride.

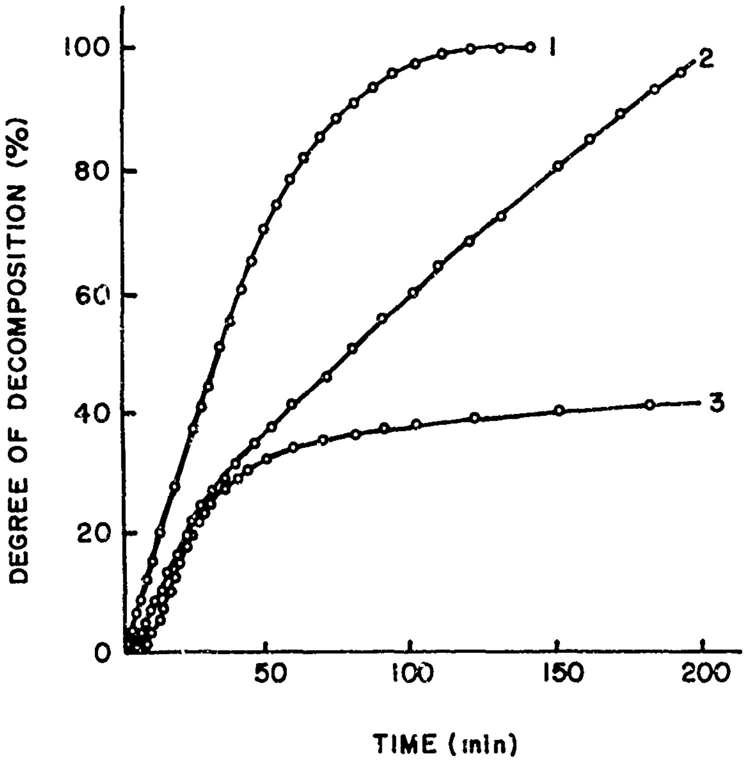
Table II lists the data on the influence of metal oxides on the thermal decomposition of the PA.

Table II. Kinetic Characteristic of the Thermal Decompositon of Ammonium Perchlorate in the Presence of Metal Oxides

Additive (5%)	Temperature Limits,* °C	Index of Degree, n	Energy of Activation, E (kcal/mole)	Preexponential Multiplier, C
MnO_2 (According to Frem[18])	208 to 229	1.6	48.2	$2.4 \cdot 10^{17}$
MnO_2 (pyrolusite)	214 to 245	1.4	33.7	$5.0 \cdot 10^{12}$
Co_2O_3	214 to 245	1.4	38.3	$7.4 \cdot 10^{14}$
CuO	239 to 270	2.2	53.5	$5.0 \cdot 10^{20}$
Cu_2O	250 to 270	1.6	45.5	$1.4 \cdot 10^{17}$
Fe_2O_3	250 to 296	1.2	41.0	$2.6 \cdot 10^{14}$
NiO	270 to 296	1.2	49.2	$6.3 \cdot 10^{17}$
V_2O_5	250 to 303	1.2	43.7	$1.6 \cdot 10^{15}$
Cr_2O_3	260 to 309	1.1	30.1	$8.0 \cdot 10^{9}$

*At a temperature above the upper limit, the mixtures burst into flame.

It is seen from Table II that the action of the additive depends on the chemical nature of the oxide. Figure 3 shows the decomposition curves of PA in the presence of some oxides.



1-MnO_2 (according to Frem), 224°C; 2-NiO, 280°C; 3-CuO, 260°C

Figure 3. Decompositon Curves of Ammonium Perchlorate in the Presence of 5 Percent by Weight of Oxides

The strongest influence on the thermal decomposition of PA is exerted by the compounds of manganese (MnO_2, $MnCO_3$, $MnCl_2 \cdot 4H_2O$) and cobalt (Co_2O_3, $CoCO_3$, CoC_2O_4, $CoCl_2 \cdot 6H_2O$). In the presence of these additives, the PA decomposes completely even at 210° to 220°C. The induction period is in most cases, absent. The rate of decomposition of PA changes practically in proportion to the content of the additive within the limits of 1 to 5 percent.

In the presence of copper compounds (Figure 3), the maximum decomposition rate increases. The degree of decomposition increases insignificantly (Figure 3). The decomposition curves of PA in the presence of copper compounds do not change their nature (the induction period at a temperature lower than 240°C is shortened.) Mixtures of PA with 5 percent $CuCO_3$, Cu_2O, CuO, and $Cu_2Cl_2 \cdot 2H_2O$ burn at temperatures of 265°, 275°, 280°, 285°, and 287°C, respectively. After the mixtures burned, a greenish scale remained on the cup, this was soluble in water, with the formation of a bluish solution, i. e., $CuCl_2$ formed. By changing the content of copper compounds within the limits of 1 to 5 percent, their influence on the decomposition of PA does not change.

Compounds of iron (Fe_2O_3, FeC_2O_4, $FeCl_3 \cdot 6H_2O$), nickel (NiO, NiC_2O_4, $NiCO_3 \cdot Ni(OH)_2$, $NiCl_2 \cdot 6H_2O$), chromium (Cr_2O_3, $CrCl_3 \cdot 6H_2O$), and vanadium (V_2O_5) influence the decomposition of PA at a temperature above 240° C.

At the same time, the maximum rate of decomposition of PA decreases, while the rate of decomposition in the third, the slowest, period increases (Figure 3). At 270° to 280° C, PA with these additives decomposes completely. Within limits of 1 to 5 percent, the rate of decomposition in the third period is proportional to the additive. The maximum rate decreases with a decrease to a certain limit in the amount of these additives. This limit amounts to about 2.5 percent for compounds of iron and nickel (II), and less than 1 percent for compounds of chromium and vanadium. With a further decrease in the amount of the additive, the maximum rate increases and approaches the maximum rate of decomposition of the pure PA. The oxides, MgO and ZnO, upon being heated, enter into chemical reaction with the PA. At the same time, a liquid phase results, which, apparently, serves to explain the influence of these additives on the decomposition of the PA.

It should be pointed out that the introduction of most tested additives strongly reduces the ignition point of the PA.

Table III lists data on the thermal decomposition of PA in the presence of different compounds of cobalt.

Table III. Kinetic Characteristics of the Decomposition of Ammonium Perchlorate in the Presence of Cobalt Compounds

Additive (5%)	Temperature Limits*, ° C	Index of Degree, n	Energy of Activation, E (kcal/mole)	Preexponential Multiplier, C
$CoCO_3$	208 to 224	1.4	46.7	$1.4 \cdot 10^{19}$
CoC_2O_4	210 to 229	1.7	43.7	$5.4 \cdot 10^{17}$
Co_2O_3	214 to 245	1.4	38.3	$7.4 \cdot 10^{14}$
$CoCl_2 \cdot 6H_2O$	214 to 250	1.1	36.0	$6.3 \cdot 10^{13}$

*At a temperature above the upper limit, the mixtures caught fire in the oven.

It is seen from Table III that the action of the additives depends on the nature of the chemical compound of the given element. The activity of the additive decreases in the following order: carbonates (oxalates), oxides, and chlorides. During the thermal decomposition of the PA, all the additives which we tested changed into oxides that are most characteristic for the given metal. The activity of the additive is apparently determined by the ease of its conversion into the oxide and the properties of the resulting oxide.

CONCLUSIONS

1) A study was made of the thermal decomposition of PA at 214° to 470°C by the gravimetric method in an air atmosphere. The following values of the energies of activation of the decomposition have been determined: orthorhombic form, E = 40 kcal/mole; cubic form, E = 24 to 28 kcal/mole; and residue from low-temperature reaction, E = 36 to 39 kcal/mole.

2) A study was made of the influence of oxides, chlorides, carbonates, and oxalates of a number of metals on the thermal decomposition of PA. It was shown that the compounds of manganese and cobalt lead to complete decomposition of the PA at a temperature below 240°C. The compounds of iron, nickel, and chromium lead to complete decomposition of PA at 270° to 280°C.

3) At the same temperature, the maximum rate of decomposition of PA with additives of the compounds of Cu, Mn, Co, as well as ZnO is greater than the maximum decomposition rate of the pure PA; the maximum decomposition rate of mixtures of PA with compounds of iron, nickel (II), chromium, as well as V_2O_5 is less than for pure PA.

4) The activity of additives of the same element decreases in the following order: carbonate (oxalate), oxide, and chloride.

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