

**INORGANIC SYNTHESIS  
AND INDUSTRIAL INORGANIC CHEMISTRY**

**Research and Development of Method  
for Potassium Acetate of High Purity**

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**Abstract**—Crystallization of potassium acetate from aqueous solutions, an effect of product yield and washing of its crystals on an efficiency of purification were investigated. Behavior of  $\text{KCH}_3\text{COO} \cdot 1.5\text{H}_2\text{O}$  was studied in heating. Based on data of the study a technological scheme of producing anhydrous potassium acetate of high purity was developed.

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Potassium acetate of pure and ultrapure grades is used as a reagent in analytical chemistry, in conducting researches, in the production of heat-resistant silicone-organic resins [1], antibiotics [2]. In recent years, potassium acetate is widely used in a fire fighting equipment and de-icing [1].

In Russia, potassium acetate is produced of analytically pure grade (State Standard GOST 5820) and of high purity 3-3 for researches (State Standard TU 6-09-4141) (Table 1).

Comparison of quality of domestic products with the best foreign samples shows that products of SIGMA BIO ULTRA (US) and FLUKA Bio Chemica Ultra (Switzerland) are of higher quality with controlling of more number of impurities (Table 2).

Scarce information on ways of potassium acetate producing is published in scientific and patent literature: obtaining by interaction of potassium hydroxide with acetic acid, acetic anhydride, solution of ammonium acetate, and also of potassium carbonate or hydrocarbonate with  $\text{CH}_3\text{COOH}$  [1–4]. It was suggested preparation of anhydrous potassium acetate from KOH of technical grade and acetic anhydride in environment of aliphatic alcohols at 40–50°C, by treatment of the obtained solution by activated carbon followed by crystallization after distillation of a solvent, filtration, and drying of the crystals. A technique was described

of preparation of anhydrous potassium acetate by interaction of KOH solution with acetic acid to pH 8.3–8.6 at 70–75°C, with purification by activated carbon

**Table 1.** Quality of potassium acetate produced in Russia

Quality indices	State Standard 5820		High purity 3-3 (State Standard TU 6-09-4141)
	analytically pure grade	pure	
Content, wt %: major substance ( $\text{CH}_3\text{COOK}$ ) in the dried product	$\geq 99.5$	$\geq 99.0$	$\geq 99.5$
substances insoluble in water	$5 \times 10^{-3}$	$1 \times 10^{-2}$	$3 \times 10^{-3}$
acidity ( $\text{CH}_3\text{COOH}$ )	$1 \times 10^{-1}$	$2 \times 10^{-1}$	$1 \times 10^{-1}$
sulfates	$2 \times 10^{-3}$	$5 \times 10^{-3}$	$2 \times 10^{-3}$
phosphates	$1 \times 10^{-3}$	$5 \times 10^{-3}$	$2 \times 10^{-4}$
chlorides	$2 \times 10^{-3}$	$5 \times 10^{-3}$	$2 \times 10^{-3}$
iron	$5 \times 10^{-4}$	$1 \times 10^{-3}$	$5 \times 10^{-4}$
calcium	$2 \times 10^{-3}$	$5 \times 10^{-3}$	$2 \times 10^{-3}$
magnesium	$1 \times 10^{-3}$	$2 \times 10^{-3}$	$1 \times 10^{-3}$
arsenic	$1 \times 10^{-4}$		$5 \times 10^{-5}$
heavy metals (Pb)	$5 \times 10^{-4}$	$1 \times 10^{-3}$	$5 \times 10^{-4}$
loss on drying	5	7	

**Table 2.** Quality of anhydrous potassium acetate produced abroad

Quality indices	Sigma Bio Ultra, USA 2008–2009	Panreak PA, Italia 2009–2010	Fluka Bio Chemica Ultra, Switzerland 2007–2008	Merck Suprapur, Germany 2008–2010	Carlo Erba for analysis, Italia
Content, wt %:					
major substance	≤99.0	≤99.0	≤99.0	≤99.0–100.5	≤99.0
substances insoluble in water		≤5 × 10 <sup>-3</sup>			≤5 × 10 <sup>-3</sup>
total nitrogen		≤1 × 10 <sup>-3</sup>			2 × 10 <sup>-3</sup> (NH <sub>4</sub> )
loss on drying				1.0 (105°C)	1.0 (130°C)
chlorides	≤5 · 10 <sup>-3</sup>	≤2 × 10 <sup>-3</sup>	≤5 × 10 <sup>-3</sup>	≤1 × 10 <sup>-2</sup>	≤1 × 10 <sup>-3</sup>
phosphates		≤1 × 10 <sup>-3</sup>			≤1 × 10 <sup>-3</sup>
sulfates	≤5 × 10 <sup>-3</sup>	≤5 × 10 <sup>-3</sup>	≤5 × 10 <sup>-3</sup>	≤5 × 10 <sup>-3</sup>	≤2 × 10 <sup>-3</sup>
aluminum	≤5 × 10 <sup>-4</sup>		≤5 × 10 <sup>-4</sup>	≤1 × 10 <sup>-4</sup>	
arsenic	≤1 × 10 <sup>-5</sup>	≤1 · 10 <sup>-4</sup>	≤1 × 10 <sup>-5</sup>	≤2 × 10 <sup>-4</sup>	≤1 × 10 <sup>-4</sup>
barium	≤5 × 10 <sup>-4</sup>		≤5 × 10 <sup>-4</sup>		
bismuth	≤5 × 10 <sup>-4</sup>		≤5 × 10 <sup>-4</sup>		
calcium	≤5 × 10 <sup>-3</sup>	≤5 × 10 <sup>-3</sup>	≤5 · 10 <sup>-3</sup>		≤5 × 10 <sup>-3</sup>
cadmium	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>		
cobalt	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>		
chromium	≤5 × 10 <sup>-44</sup>		≤5 × 10 <sup>-4</sup>		
copper	≤5 × 10 <sup>-44</sup>	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>		≤1 × 10 <sup>-4</sup>
iron	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>	≤1 × 10 <sup>-3</sup>	≤5 × 10 <sup>-4</sup>
sodium	≤0.5	≤0.25	≤5.0	≤0.5	≤2 × 10 <sup>-3</sup>
lithium	≤5 × 10 <sup>-4</sup>		≤5 × 10 <sup>-4</sup>		
magnesium	≤1 × 10 <sup>-3</sup>	≤5 × 10 <sup>-3</sup>	≤1 × 10 <sup>-3</sup>		≤5 × 10 <sup>-3</sup>
manganese	≤5 × 10 <sup>-4</sup>		≤5 × 10 <sup>-4</sup>		
molybdenum	≤5 × 10 <sup>-4</sup>		≤5 × 10 <sup>-4</sup>		
nickel	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>		≤2 × 10 <sup>-4</sup>
lead	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>	≤4 × 10 <sup>-4</sup>	≤3 × 10 <sup>-4</sup>
strontium	≤5 × 10 <sup>-4</sup>		≤5 × 10 <sup>-4</sup>		
zinc	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>	≤5 × 10 <sup>-4</sup>	≤4 × 10 <sup>-4</sup> (Hg)	≤2 × 10 <sup>-4</sup>
pH of 1 M solution	7.0–9.0	6.5–9.0	7.0–9.0	7.5–8.5	7.5–8.0

followed by crystallization in the presence of tartaric acid (0.005–0.025 wt %), filtration, and drying of the crystals [1]. Also production of anhydrous potassium acetate is known by treatment of potassium carbonate or bicarbonate powder by ice CH<sub>3</sub>COOH taken with a 2.5% excess in term of stoichiometry with stirring followed by heating the reaction mixture to 110–150°C

[4]. All described techniques can provide the products of reactive grade.

Among the techniques well-known from the literature for preparation of especially pure substances with regard to obtaining of potassium acetate the crystallization from aqueous solutions can be of practical interest [5]. However, no research on the use of the technique of

**Table 3.** Solubility of potassium acetates and accompanying elements in water [6]

Compound	Solubility, wt%, at temperature, °C							
	0	10	20	30	50	60	80	90
KCH <sub>3</sub> COO·1.5H <sub>2</sub> O	68.4	70.0	71.9	73.9	76.4 (40°C)			
Mg(CH <sub>3</sub> COO) <sub>2</sub> ·4H <sub>2</sub> O	36.2	38.0 (15°C)	39.3 (25°C)	41.8 (35°C)	49.8 (55°C)			
Ca(CH <sub>3</sub> COO) <sub>2</sub> ·2H <sub>2</sub> O	27.2	26.5	25.8	25.3		24.6	25.1	23.7
Sr(CH <sub>3</sub> COO) <sub>2</sub> ·0.5H <sub>2</sub> O	27.0	30.4	29.5 (15°C)	28.7 (25°C)	27.2	26.6 (70°C)	26.5	26.6
Ba(CH <sub>3</sub> COO) <sub>2</sub> ·3H <sub>2</sub> O	37.0	40.9 (18 °C)	43.3 (26°C)	42.8	43.5	42.8	42.5	42.8 (99°C)
Pb(CH <sub>3</sub> COO) <sub>2</sub> ·3H <sub>2</sub> O	16.5	22.8	30.7	41.1	67.6			
NaCH <sub>3</sub> COO·3H <sub>2</sub> O	26.6	29.0	31.7	35.3	45.4	58.2	60.5	61.7

obtaining high purity potassium acetate was found in the literature.

Potassium acetate is highly soluble in water, has a positive temperature coefficient of solubility [6], its crystallization from aqueous solutions may be promising for obtaining a product of high purity.

For forecasting the effectiveness of the crystallization purification of potassium acetate we reviewed published data on phase equilibria in water–salt systems with its participation and with acetates of accompanying elements.

Analysis of KCH<sub>3</sub>COO–M<sup>n+</sup>(CH<sub>3</sub>COO)<sub>n</sub>–H<sub>2</sub>O systems (M<sup>n+</sup> is Co<sup>2+</sup> [7, 8], Ni<sup>2+</sup> [9], Cu<sup>2+</sup> [10]) at 25°C indicates the absence of iso- and isodimorphism of components. In the crystallization region of potassium acetate in the systems considered [7–10] the formation of solid solutions was not observed, and, therefore, copper acetate(II), cobalt(II), nickel(II) will be concentrated in the mother liquor. Data on the study of water–salt acetate systems with K–Fe, K–Mg, K–Ca, K–Sr, K–Ba in the literature were not found. Large solubility of acetates of accompanying elements in water (Table 3) points to the possibility of their separation in the crystallization region of potassium acetate (concentration in mother liquor).

Literature data on structures of potassium acetate and accompanying elements (Table 4) demonstrate that KCH<sub>3</sub>COO·1.5H<sub>2</sub>O is crystallized in tetragonal system, in its crystallization lead(II, IV), calcium, magnesium, zinc, copper(II), cobalt (II), nickel (II), sodium acetate

**Table 4.** Crystal structures of potassium acetate and a number of accompanying elements

Compound	Type of crystal lattice	Reference
KCH <sub>3</sub> COO·1.5H <sub>2</sub> O	Tetragonal, space group P4/cc	[11]
Pb(CH <sub>3</sub> COO) <sub>4</sub>	Monoclinic, space group P2 <sub>1</sub> /c	[12]
Pb(CH <sub>3</sub> COO) <sub>2</sub> ·3H <sub>2</sub> O	Monoclinic, space group C2/m	[13]
Zn(CH <sub>3</sub> COO) <sub>2</sub> ·2H <sub>2</sub> O	Monoclinic, space group C2/c	[14]
Mg(CH <sub>3</sub> COO) <sub>2</sub> ·4H <sub>2</sub> O	Monoclinic, space group P2 <sub>1</sub> /c	[15]
Ca(CH <sub>3</sub> COO) <sub>2</sub> ·2H <sub>2</sub> O	Triclinic, space group P1	[16, 17]
Co(CH <sub>3</sub> COO) <sub>2</sub> ·4H <sub>2</sub> O	Monoclinic, space group P2 <sub>1</sub> /c	[18]
Cu(CH <sub>3</sub> COO) <sub>2</sub> ·H <sub>2</sub> O	Monoclinic, space group C2/c	[19]
Ni(CH <sub>3</sub> COO) <sub>2</sub> ·4H <sub>2</sub> O	Monoclinic, space group P2 <sub>1</sub> /c	[18]
NaCH <sub>3</sub> COO·3H <sub>2</sub> O	Monoclinic, space group C2/c	[20]

present in the solution will be concentrated in the mother liquor and their capture by crystals can be due to occlusion and adsorption.

In view of the above the crystallization of potassium acetate from aqueous solutions was investigated experimentally. The effect of the output of potassium acetate and washing of its crystals on the efficiency of the purification was examined.

## EXPERIMENTAL

Available reagents: acetic acid (analytically pure, State Standard GOST 61) and potassium hydroxide (reagent grade, GOST 24363) were used as an original raw material for potassium acetate preparation. Potassium acetate (analytically pure, GOST 5820) is produced in Russia in small amounts as a custom reagent, and its use is not appropriate as an original raw material for obtaining the product of high purity.

The calculated amount of KOH crystals (reagent grade) was charged in a glass apparatus equipped with an anchor stirrer and electric heating, and then acetic acid (analytically pure) with an excess of 30 wt % in term of stoichiometry was slowly added. The resulting solution of potassium acetate was filtered off from possible mechanical impurities by paper filter "blue ribbon," a sample was taken for analysis, the solution was evaporated at  $115 \pm 10^\circ\text{C}$  to a density of  $1.38\text{--}1.41 \text{ g cm}^{-3}$ . Afterwards the contents was cooled to

$25^\circ\text{C}$  with stirring, the crystals  $\text{KCH}_3\text{COO}\cdot1.5\text{H}_2\text{O}$  were separated from the mother liquor, and a sample was taken for analysis. The remaining crystals were washed on the funnel with a saturated solution of potassium acetate of high purity (200 ml per 100 g of crystals: the ratio was chosen experimentally) and thoroughly wrung out.

The sample of the solution for the analysis (the original) was evaporated to dryness, the samples of crystals before and after washing were dried and the content of impurities was determined.

The samples were examined by atomic emission method with excitation in an inductively coupled plasma (plasma spectrometer Baird, USA) and preconcentration of impurities. A determination error was 10–30% depending on properties of an impurity and its content [21].

Based on the results we calculated purification coefficients  $K_{\text{pur}}$  as a ratio of impurity content before and after crystallization (washing). Crystallization degree  $\alpha$  was 0.8 [5].

The data of Table 5 show that in the crystallization of  $\text{KCH}_3\text{COO}\cdot1.5\text{H}_2\text{O}$  the purification from the majority of the impurities ( $K_{\text{pur}} > 3\text{--}50$ ) occurs. The value of  $K_{\text{pur}}$  of  $\text{KCH}_3\text{COO}\cdot1.5\text{H}_2\text{O}$  crystals after washing for most of these impurities could not be found due to the insufficient sensitivity of the analysis method.

As a whole, these data confirm the assumption on usefulness of the crystallization for purification of po-

**Table 5.** Results of analysis of laboratory samples of potassium acetate

Impurity	Content of impurity, wt %, in the sample			Purification coefficient, $K_{\text{pur}}$
	original	after crystallization without washing	after crystallization with washing	
Iron	$7 \times 10^{-5}$	$4 \times 10^{-5}$	$1 \times 10^{-5}$	7
Cobalt	$3 \times 10^{-6}$	$1 \times 10^{-6}$	$<1 \times 10^{-6}$	$> 3$
Manganese	$5 \times 10^{-5}$	$2 \times 10^{-5}$	$<1 \times 10^{-5}$	$> 5$
Nickel	$5 \times 10^{-5}$	$1 \times 10^{-5}$	$<1 \times 10^{-6}$	$> 50$
Copper	$1 \times 10^{-5}$	$1 \times 10^{-6}$	$<1 \times 10^{-6}$	$> 10$
Chromium	$7 \times 10^{-5}$	$5 \times 10^{-5}$	$2 \times 10^{-5}$	$> 3$
Magnesium	$3 \times 10^{-4}$	$1 \times 10^{-5}$	$<1 \times 10^{-5}$	$> 30$
Calcium	$5 \times 10^{-4}$	$<1 \times 10^{-4}$	$<1 \times 10^{-4}$	$> 5$
Strontium	$3 \times 10^{-5}$	$1 \times 10^{-5}$	$5 \times 10^{-6}$	6
Barium	$8 \times 10^{-5}$	$5 \times 10^{-5}$	$3 \times 10^{-6}$	25
Lead	$4 \times 10^{-5}$	$2 \times 10^{-6}$	$<2 \times 10^{-6}$	$> 20$
Sodium	$2 \times 10^{-1}$	$1 \times 10^{-1}$	$2 \times 10^{-2}$	10

tassium acetate. Comparison of the crystal structures  $\text{KCH}_3\text{COO}\cdot 1.5\text{H}_2\text{O}$  [11] and acetate of accompanying elements [12–20] (Table 4) with the data after research of the systems  $\text{KCH}_3\text{COO}-\text{M}^{n+}(\text{CH}_3\text{COO})_n-\text{H}_2\text{O}$  ( $\text{M}^{n+} = \text{Co}^{2+}, \text{Ni}^{2+}, \text{Cu}^{2+}$ ) at 25°C [7–10], the solubility of a number of acetates (Table 3) points to the effectiveness of the purification of potassium acetate by crystallization with an additional washing of the crystals. The impurities of the accompanying elements are concentrated in the mother liquor, and their capture by crystals  $\text{KCH}_3\text{COO}\cdot 1.5\text{H}_2\text{O}$  may be due to occlusion (adsorption is excluded under the terms of the experiment as the excess of  $\text{CH}_3\text{COOH}$  is introduced).

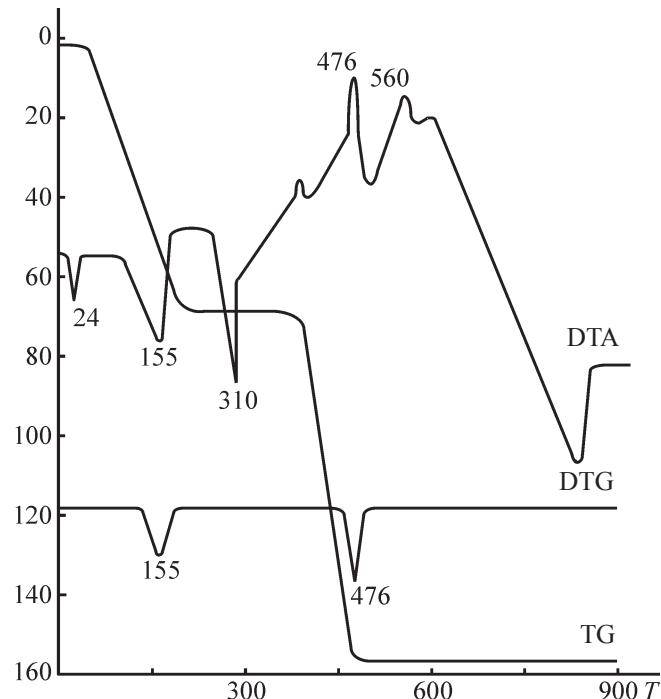
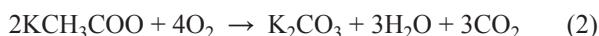
For selection of the drying conditions of crystalline hydrate of potassium acetate in the process of obtaining anhydrous product of high purity a behavior of  $\text{KCH}_3\text{COO}\cdot 1.5\text{H}_2\text{O}$  was investigated in heating (a derivatograph MOM, Hungary). The heating was carried out in air up to 1000°C at a rate of heating  $10^\circ \text{ min}^{-1}$  at atmospheric pressure and humidity, temperature measurement accuracy was  $\pm 5^\circ$ . Reference  $\text{Al}_2\text{O}_3$  calcined at 1000°C. The weighed sample in an alundum crucible was 300–400 mg.

According to data of thermogravimetric investigation the compound  $\text{KCH}_3\text{COO}\cdot 1.5\text{H}_2\text{O}$  was stable in heating to 35–40°C, at higher temperatures it was decomposed through two stages (Fig. 1). On the DTA curve an endothermic effect was recorded at 24°C that is consistent with published data on the phase transition [22, 23]. In the temperature range 40–250°C the compound has lost 1.5 molecules of crystallization water, the weight loss was 19.51% (theoretical 21.57%):



In this case there is a fairly large endothermic effect on the diagram at  $T \sim 155^\circ\text{C}$ , which is a superposition of two effects of the phase transition known in the literature [22, 23] and crystalline decomposition. Horizontal sections on the curves TG, DTG in the temperature range 232–364°C are evidence of the stability of the resulting anhydrous compound  $\text{KCH}_3\text{COO}$ . The endothermic effect at  $T \sim 310^\circ\text{C}$  corresponds to melting of  $\text{KCH}_3\text{COO}$  and is consistent with the data of [9, 24, 25].

The second stage of the sample weight loss in the temperature range 340–520°C corresponds to the decomposition to potassium carbonate of  $\text{KCH}_3\text{COO}$  formed in the first stage:

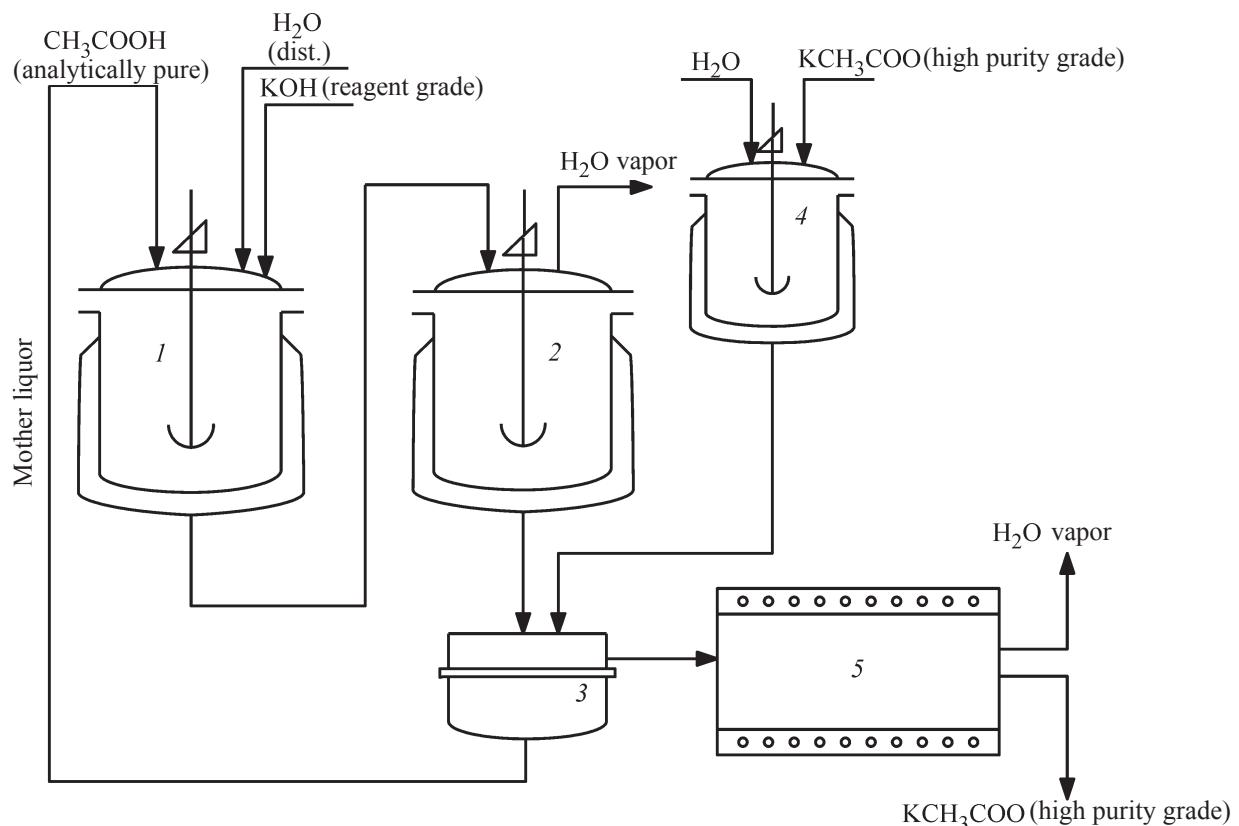


**Fig. 1.** DTG, DTA, TG curves for  $\text{KCH}_3\text{COO}\cdot 1.5\text{H}_2\text{O}$ . ( $\Delta m$ ) Weight loss, %; ( $T$ ) temperature, °C.

and practically coincides with the literature data [9, 24, 25]. In contrast to results of [9, 22, 24, 25] we did not find the formation of potassium oxalate as an intermediate. In the region of  $\text{K}_2\text{C}_2\text{O}_4$  decomposition (500–600°C) on the derivatogram there is no transformation associated with the weight loss.

Based on the research we developed the technological scheme of the producing anhydrous potassium acetate of high purity (Fig. 2). The major stages of the process were: (1) obtaining a solution of potassium acetate, (2) evaporating the solution, crystallization with stirring; (3) separation of crystals  $\text{KCH}_3\text{COO}\cdot 1.5\text{H}_2\text{O}$ , washing them with its saturated solution of high purity prepared in the reactor (4), (5) drying. The evaporation of solution of  $\text{KCH}_3\text{COO}$  was carried out to the density of  $1.38\text{--}1.41 \text{ g cm}^{-3}$  at  $115 \pm 10^\circ\text{C}$ , the content was cooled with stirring to 25°C, the precipitated crystals of  $\text{KCH}_3\text{COO}\cdot 1.5\text{H}_2\text{O}$  were carefully separated from the mother liquor, washed with a saturated solution of potassium acetate of high purity ( $1 : s = 1 : 2$ ), and dried in a shelf drying box at  $150 \pm 5^\circ\text{C}$ . The mother liquor from the  $\text{KCH}_3\text{COO}\cdot 1.5\text{H}_2\text{O}$  crystallization stage was directed at the stage (1) of obtaining the solution.

Comparison of the results of laboratory analysis of the sample shows that by the developed technique the



**Fig. 2.** Technological scheme for production of  $\text{KCH}_3\text{COO}$  of high purity. (1) Reactor for preparation of  $\text{KCH}_3\text{COO}$  solution, (2) evaporator, (3) a filter for separating and washing the crystals  $\text{KCH}_3\text{COO} \cdot 1.5\text{H}_2\text{O}$ , (4) reactor for preparation of the washing liquid, (5) shelf drying box.

**Table 6.** Quality of the laboratory sample of potassium acetate and the best foreign analogues (by catalogs)

Indices	Laboratory sample	Sigma Bio Ultra, USA	Fluka BioChemica Ultra, Switzerland
Content, wt %: major substance	99.5	99.0	99.0
iron	$1 \times 10^{-5}$	$5 \times 10^{-4}$	$5 \times 10^{-4}$
vanadium	$<1 \times 10^{-6}$		
cobalt	$<1 \times 10^{-6}$	$5 \times 10^{-4}$	$5 \times 10^{-4}$
manganese	$<1 \times 10^{-5}$	$5 \times 10^{-4}$	$5 \times 10^{-4}$
copper	$<1 \times 10^{-6}$	$5 \times 10^{-4}$	$5 \times 10^{-4}$
nickel	$<1 \times 10^{-6}$	$5 \times 10^{-4}$	$5 \times 10^{-4}$
chromium	$2 \times 10^{-5}$	$5 \times 10^{-4}$	$5 \times 10^{-4}$
magnesium	$<1 \times 10^{-5}$	$1 \times 10^{-3}$	$1 \times 10^{-3}$
calcium	$<1 \times 10^{-4}$	$5 \times 10^{-3}$	$5 \times 10^{-3}$
lead	$<5 \times 10^{-5}$		
sodium	$2 \times 10^{-2}$	0.5	5.0

anhydrous potassium acetate is obtained of very high purity and it is of much higher quality than the best foreign analogues (Table 6).

## CONCLUSIONS

- (1) The high efficiency of the purification of potassium acetate by crystallization from aqueous solutions was demonstrated.
- (2) The behavior of  $\text{KCH}_3\text{COO} \cdot 1.5\text{H}_2\text{O}$  in heating was studied.
- (3) The technological scheme for production of anhydrous potassium acetate of high purity was developed.

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