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Research on Na-Monochloroacetate Production Processes Based on Local Raw Materials

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A B S T R A C T
The main application of monochloroacetic acid is the production of carboxymethylcellulose (CMC). Up to 30% of the global volume of MCA is spent on this. Carboxymethylcellulose is one of the components of thickeners for wallpaper glue, detergents, soaps and emulsion paints. Ceramics production is also not complete without carboxymethylcellulose - in this industry it is used as a thickener, plasticizer, binder and agent for increasing the smoothness of enamels. In the food industry, this modification of cellulose is known by the abbreviation E466 and acts as a thickener, emulsifier and stabilizer.

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Monochloroacetic acid is also necessary for agrochemical enterprises. In agrochemistry, it acts as an intermediate in the production of active ingredients. The latter, in turn, are the main components of agrochemicals for the protection of plant plants, such as herbicides or insecticides. Monochloroacetic acid is used to produce the following substances:

- acid 2,4-D (2,4-dichlorophenoxyacetic acid),
- dithiophosphoric acid esters (e.g. dimethoate),
- Chloroacetyl chloride (CAC),
- Trichloroacetyl chloride (TCAC),
- 2,4,5-trichlorophenoxyacetic acid (2,4,5-T),
- 2-methyl-4-chlorophenoxyacetic acid (MCPA),
- Phosphonates (e.g. glyphosate, the main active ingredient in the widely used herbicide Roundup).

The importance of monochloroacetic acid for manufacturers of cosmetics and personal care products should not be overlooked, as it is essential for the production of betaines, amphoteric surfactants with foaming properties, which are used in shampoos.[64] Thioglycolic acid (THC or mercaptoacetic acid), which is found in hair perms, is also produced using monochloroacetic acid.

Monochloroacetic acid is an intermediate in the synthesis of indigo and many other dyes; it is used in the production of carboxymethylcellulose, the hypnotic barbital, herbicides (e.g., salts and esters of 2,4-dichlorophenoxyacetic acid), and vitamin B6.

Its utility in organic chemistry is also in the O-alkylation of salicylaldehyde with chloroacetic acid, followed by decarboxylation of the resulting ester to give benzofuran.

Basically, monochloroacetic acid is obtained by chlorination of glacial acetic acid under catalysis with acetic anhydride.

$CH_3COOH+Cl \rightarrow CH_2ClOOH+HCl\uparrow$

Another industrial method is the hydrolysis of trichloroethylene

$CCl_2CHCl+2H_2O \rightarrow CH_2ClOOH+ 2HCl\uparrow$

Hydrolysis gives a pure product, while chlorination requires complex distillation to separate mono-, bi-, and trichloroacetic acids. Annual production of chloroacetic acid is approximately 420,000 tons. When chloroacetic acid reacts with acetic acid in the presence of a small amount of red phosphorus, a mixture of mono-, di-, and trichloroacetic acids is formed.

At the next stage, the processes for obtaining Na-monochloroacetate from monochloroacetic acid were studied.

Technical sodium monochloroacetate (TU 2432-241-05763458-98) is the sodium salt of monochloroacetic acid. Sodium monochloroacetate is used as a component in the production of herbicides. Sodium monochloroacetate is also produced by spraying dissolved monochloroacetic acid (MSA) with a 50% sodium hydroxide solution in a sprayer.

Chemical formula of sodium monochloroacetate:

CH2CICOONa

Appearance: white powder

PROPERTIES:

Sodium monochloroacetate mass fraction minimum 93%

Sodium dichloroacetate mass fraction maximum 2%

Chloride mass fraction (for sodium chloride) maximum 1.5%

Alkalinity by sodium carbonate, 0.1-1.0%

Water mass fraction, 3% maximum

pH in a 1% aqueous solution 5.5-10

Table 1. Technical characteristics of sodium monochloroacetate (TU 2432-241-05763458-98)

Ko‘rsatkichlar	**Norma**	
	top grade	**first grade**
Appearance	White powder	White powder
Mass fraction of sodium monochloric acid, %, not less	95	93
Mass fraction of dichloroacetic acid, %, not more than	1,5	2
Mass fraction of chlorides (for sodium chloride),%, not more	1,2	1,5
Alkalinity by sodium carbonate, %	0,1-0,5	0,1-1,0
Mass fraction of water, %, not more	2	3
pH in a 1% aqueous solution	5,5-5,7	5,5-10

Various fractions of Na-monochloroacetate were obtained by reacting sodium hydroxide with soda ash. The table below shows some of the quality parameters of various fractions of NaMXA (Na-monochloroacetate) intended for the production of Na-KMS and PATS of different grades.

The reaction equation is:

$2ClCH_2COOH+Na_2CO_3=2ClCH_2COONa+H_2O+CO_2$

Table 2. Effect of increasing Na2CO3 consumption rate on the quality parameters of the resulting NaMXA (Na-monochloroacetate)

№	Reogentlar		NaMXA quality indicators		
	$CH_2ClCOOH$ kg	Na_2CO_3 kg	Mass fraction of sodium monochloric acid,%	Mass fraction of water, %,	pH
1	189	106 (NaMXA-1)	98	3	6,4
2		111 (NaMXA-2)	95	2	6,1
3		116 (NaMXA-3)	89	3	5,8
4		121 (NaMXA-4)	85	4	5,5
5		126 (NaMXA-5)	78	6	5

From the table, it is possible to observe the effect of increasing the Na2CO3 consumption rate on the quality indicators of the resulting NAMXA (Na-monochloroacetate). In this case, an increase in Na2CO3 leads to a decrease in the mass fraction of sodium monochloric acid. This prevents the synthesis of Na-KMS and PATS from obtaining their quality indicators, that is, acceptable grades.

Analysis of some quality indicators of the obtained Na-monochloroacetate by physicochemical and mechanical-structural properties

Table 3. Comparison of the quality parameters of Na-monochloroacetate with the technical parameters of sodium monochloroacetate (TU 2432-241-05763458-98)

Ko‘rsatkichlar	Standard quality indicators according to TU		Standard quality indicators by fractions				
	top grade	first grade	NaMXA1	NaMXA2	NaMXA3	NaMXA4	NaMXA-5
Appearance	White powder	White powder	White powder				
Mass fraction of sodium monochloric acid, %, not less	95	93	98	95	89	85	78
Mass fraction of dichloroacetic acid, %, not more	1,5	2	-	-	-	-	-
Mass fraction of chlorides (for sodium chloride), %, not more	1,2	1,5	-	-	-	-	-
Alkalinity by sodium carbonate, %	0,1-0,5	0,1-1,0	-	-	-	-	-
Mass fraction of water, %, not more	2	3	3	2	3	4	6
pH in a 1% aqueous solution	5,5-5,7	5,5-10	6,4	6,1	5,8	5,5	5

Analysis

10.00g of the drug is placed in a 100 cm^3 conical flask, dissolved in 50 cm^3 of water and three drops of phenolphthalein solution are added to it. If the solution remains colorless, then sodium hydroxide solution is added dropwise from the burette until a pink color appears. If the solution is colored pink, then hydrochloric acid solution is added dropwise from the burette until the pink color disappears.

- The mass fraction of acids in acetic acid (CH_3COOH) or the mass fraction of alkalis in sodium hydroxide (NaOH) (X) in percent is calculated by the formula.

where is the volume of sodium hydroxide solution with a molar concentration of 0.1 mol/dm3 or hydrochloric acid solution with a molar concentration of exactly 0.1 mol/dm3 of the amount spent for titration, cm3;

- m is the mass of the drug sample, g;
- m1 - mass of CH3COOH (0.006) or NaOH (0.004), 1 cm3 of sodium hydroxide solution with a molar concentration of exactly 0.1 mol/dm3 or hydrochloric acid solution with a molar concentration of exactly 0.1 mol/dm3, g. .
- The analysis result is taken as the arithmetic mean of the results of two parallel determinations, the absolute discrepancy between which does not exceed the permissible difference equal to 0.004%.

The permissible absolute total error of the analysis result is ±0.002% with a confidence probability P =0.95.

3.5. Determination of the mass fraction of sulfates

The determination is carried out in accordance with GOST 10671.5. In this case, 2.00 g of the drug is placed in a conical flask with a capacity of 50 or 100 cm3 (with a mark of 25 cm3) and dissolved in 15 cm^3 of water. Then 0.1 cm^3 of a 0.1% mass fraction of 2,4-dinitrophenol solution (prepared according to GOST 4919.1) is added dropwise and, stirring constantly, until the solution changes color. If the solution is cloudy, filter it through an ashless blue ribbon filter washed with hot water. Then the volume of the solution is brought to 25 cm3 with water, and then the determination is carried out using a phototurbidimetric or visual nephelometric method (method 1).

The drug is considered to comply with the requirements of this standard if the mass of sulfates does not exceed:

for the pure drug for analysis - 0.02 mg,

for the pure drug - 0.04 mg.

In case of disagreement in assessing the mass fraction of sulfates, the analysis is carried out using the phototurbidimetric method.

3.6. Determination of the mass fraction of phosphates

The determination is carried out in accordance with GOST 10671.6. In this case, 10.00 g of the drug is placed in a cylinder with a capacity of 50 cm with a soil stopper (GOST 1770) and dissolved in 7.5 cm of nitric acid solution with a mass fraction of 50%. The volume of the solution is brought to 15 cm3 with water, mixed, and then determined photometrically by the yellow color of the phosphovanadium-molybdenum complex.

The drug is considered to meet the requirements of this standard if the mass of phosphates does not exceed:

for a pure drug for analysis - 0.02 mg,

for a pure drug - 0.10 mg.

It is allowed to complete the determination visually by applying a phosphovanadium molybdenum complex to a layer of isoamyl alcohol (5 cm3).

At the same time, a control experiment is carried out under the same conditions and with the same amount of reagents. If phosphate impurities are detected, the analysis result is adjusted.

If there is a disagreement in assessing the mass fraction of phosphates, the determination is completed photometrically.

3.7. Determination of the mass fraction of chlorides

The determination is carried out in accordance with GOST 10671.7. In this case, 2.00 g of the drug is placed in a volumetric flask (when determined by the phototurbidimetric method) or a conical flask with a capacity of 100 cm, marked with 40 cm (when determined by the visual nephelometric method) and dissolved in 30. cm of water. Then 3.5 cm of a 25% nitric acid solution is added, heated to boiling and cooled. If the solution is turbid, it is filtered through an ashless “blue ribbon” filter, previously washed with a hot 1% nitric acid solution. Further, the determination is carried out using the phototurbidimetric or

visual nephelometric method, without adding nitric acid solution.

The drug is considered to comply with the requirements of this standard if the mass of chlorides does not exceed:

for a pure drug for analysis - 0.020 mg,

for a pure drug - 0.020 mg.

At the same time, a control experiment is carried out under the same conditions and with the same amount of reagents. If chloride impurities are detected, the analysis result is adjusted.

If there is a disagreement in assessing the mass fraction of chlorides, the analysis is carried out using the phototurbidimetric method.

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