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CHARACTERISTICS OF REGENERATION OF N-METHYLPYRROLIDONE

G. A. Rudova and I. Z. Éifer

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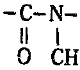
In the search for solvents less toxic than amide solvents, investigators have turned their attention to N-methylpyrrolidone (N-MP). This is probably because N-MP has many common properties with amide solvents: their high dissolving power increases when a salt component is added to the solvent; in addition, N-MP forms complexes with many salts. There are N-MP complexes with lithium chloride, perchlorate, bromide, and cyanide; nickel chloride, bromide, and iodide; cobalt chloride and bromide, and others. These complexes of the nR—salt or nR—H_2O—salt crystal solvate type (where R is a solvent) can be separated from saturated solutions during cooling. The complexing power of N-MP with salts and the possibility of separating these complexes as a crystalline product creates grounds for using the method described in [1, 2] in regeneration of N-MP and salt. This method is based on crystallization of a complex salt from a saturated solution of the salt in a solvent and rectification of the mixture of precipitator and solvent.

SEPARATION OF N-MP FROM AQUEOUS MIXTURES BY RECTIFICATION

Deep vacuum rectification is obligatory in separating the precipitator—solvent mixture for some solvents, while atmospheric rectification can be used for others. This is basically a function of the resistance of the solvents to heat and mass exchange.

N-MP turns yellow when heated and coffee brown when heated for a long time. According to the data from BASF (Germany) [3], yellowness of the product does not imply a change in the chemical purity of N-MP. However, it is only possible to agree with this if the N-MP is either used as a solvent for polymers or in synthesis, where there are no rigorous requirements for impurities in the solvent. The yellow and brown color of N-MP is probably the result of its resinification. The analysis of domestic and foreign published data on the properties of N-MP showed that polyamide resins are possible products of resinification of N-MP [4, 5]. This is indicated by the 3-methylaminobutyric acid formed in hydrolysis of N-MP (breaking of the  $-\underset{\underset{O}{\|}}{C}-\underset{\underset{CH_3}{|}}{N}-$ bond).

Even with gentle heating, this acid can form a di-, tri-, tetra-, and polymer. We also know [6] that N-MP is hydrolyzed by a strong acid or base with formation of 3-methylbutyric acid with a rate that is a function of the temperature and pH of the medium. The concentration of N-MP decreases by $2 \cdot 10^{-3}\%$ as a result of hydrolysis in an aqueous solution of N-MP at 100°C for 1 h at pH = 5, and it decreases by $2 \cdot 10^{-2}\%$ at pH = 9. The behavior of the solvent in heat and mass exchange is greatly dependent on the purity of the starting product.

Our chromatographic analysis of the composition of N-MP showed that the domestic commercial product contains 7 impurities corresponding to the following retention times: 1.2, 1.5, 2.1, 2.4, 3.3, 3.7, and 6.5 min (Fig. 1a). The retention time of N-MP is 4.1 min.

Conditions of analysis: argon carrier gas, flame-ionization detector, 270°C evaporation temperature, 220°C column thermostat temperature, 1.10 m/sec hydrogen and carrier gas flow rate, 1.10 m/sec air flow rate, Chezosorb AW sorbent with 0.20-0.36 mm particle size impregnated with 15% Carbowax 1500, 3000 × 4 mm metal columns.

Of the indicated impurities, only Nos. 1 and 2 (1 — γ-butyrolactone, 2 — water) were identified. Acids (methylamidohydroxybutyric, γ-methylaminobutyric, succinic), succinimide, furfural, etc., are possible impurities.

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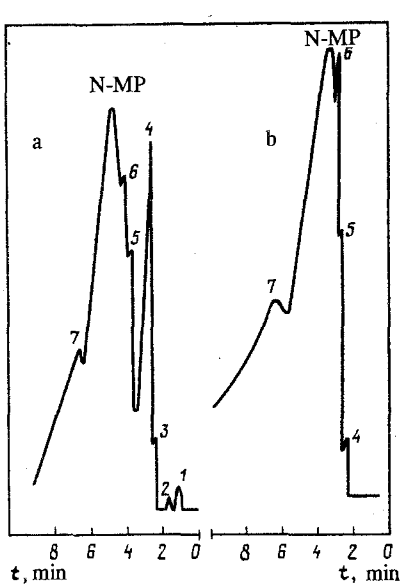

Fig. 1

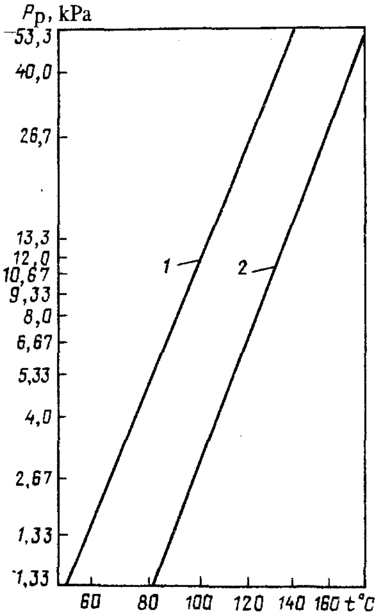

Fig. 2

Fig. 1. Chromatograms of commercial N-MP (a) and vapor phase of rectificate of N-MP (b).

Fig. 2. Partial saturated pressure P_p vs. temperature t in using DMAA (1) and N-MP (2) solvents.

Methylamidohydroxybutyric acid is an intermediate product of synthesis of N-MP; γ-methylaminobutyric and succinic acids, furfural, and succinimide could arise as a result of photolysis, hydrolysis, thermal degradation, and oxidation of N-MP.

The resistance of N-MP to heat and mass exchange, particularly during rectification, were evaluated by qualitative analysis of the product obtained for the number of impurities and the relative increase or decrease in impurities in comparison to the commercial product. A sample of bottoms product was completely analyzed, since the conditions of the stay of N-MP in the bottoms product are characterized by a higher temperature, longer stay, and maximum concentration of products of decomposition [7, 8].

Rectification was conducted on a domestic laboratory installation (30 mm column diameter, 600 mm packing height, packing: metal coil sections). Operating conditions: total recycling of bottoms, amount of vapor phase close to vapor phase in "flooding" mode at absolute pressure of 5-13 kPa, concentration of water in initial solution: from insignificant amount ("traces") to 10%, duration: from 1 to 8 h. The rectification temperature varied from 100 to 140°C. The sample of bottoms product was collected in the form of liquid and vapor phase.

The results of the study showed that in sampling the product from the column still, impurity No. 1 is virtually absent in the regenerated N-MP in all conditions; impurity No. 3 is present as "traces," similar to commercial N-MP; impurity No. 4 in the aqueous phase increases with an increase in the temperature and duration of the process and is virtually absent in insignificant amounts of water and for all temperature conditions; impurity No. 5 is present in all samples, similar to commercial N-MP; the concentration of impurity No. 6 increases significantly with a 5-10% concentration of water, and the concentration of impurity is similar to the commercial product in the other conditions; the concentration of impurity No. 7 increases in anhydrous medium with an increase in the temperature. Impurity No. 4 is probably a product of hydrolysis of N-MP, while impurity No. 7 is related to thermal degradation of N-MP.

In these conditions of rectification and sampling of the target product from the column still in the form of vapor phase, the quality of the regenerated N-MP is higher than for the commercial product: it does not contain impurities Nos. 1-3, and the concentration of impurities Nos. 4-7 is significantly reduced. The lowest concentration of these impurities in the regenerated N-MP was observed at 100-115°C (Fig. 1b).

The parameters of rectification of aqueous solutions of N-MP and dimethylacetamide (DMAA) were comparatively evaluated in the second stage of the studies. The number of theoretical plates (NTP) required for total separation of water and solvent in both cases was graphically calculated with the experimental data from a number of investigators on vapor—liquid equilibrium for N-MP—H_2O [9] and DMAA—H_2O mixtures [10] with respect to the properties of the rectificates (concentration of water no greater than 0.05%) and distillates (concentration of the corresponding solvents no greater than 0.05%). Seven theoretical plates are required for separation of DMAA—H_2O mixture, and six are required for N-MP—H_2O mixture. The distribution of the number of plates between the rectifying and stripping sections of the column as a function of the composition of the feed was examined by the method of graphic calculation of NTP. For the mixture containing a 50% concentration of solvent, there should be no more than two theoretical plates in the rectifying section and no more than 4 theoretical plates in the stripping section in separation of N-MP—H_2O mixture.

Rectification separation of DMAA—H_2O and N-MP—H_2O mixtures in identical conditions on a laboratory installation was comparatively evaluated simultaneously with the theoretical calculation of the NTP.

The study was conducted with the following method. The working flask (still) was filled with 400-450 ml of the investigated solution. When stable conditions were obtained, a sample of distillate and bottoms product was collected. The concentration of water in the initial and bottoms products was determined by potentiometric titration with Fischer's reagent, and the concentration of solvent in the distillate was determined with the photocolorimetric method in [11, 12].

The analysis of the results obtained confirmed that a smaller number of separation steps is required for separation of the N-MP—H_2O mixture than for the DMAA—H_2O mixture (30-40 analogous sieve-valve plates). Based on an industrial experiment in rectification separation of DMAA—H_2O mixture in columns with sieve-valve plates (0.24 kPa plate strength), it was found that 40-50 real separation steps (feed to 20, 24, 28 plates, counting down) are necessary for ensuring the required composition of the distillate (distillation water) and rectificate of DMAA. Temperature conditions in which accumulation of impurities in the column still as a result of hydrolysis and thermal degradation of DMAA will be minimum are thus required (maximum temperature of under 120°C in the column still, which corresponds to an absolute pressure in the column still of less than 20 kPa).

It follows from the results of the study of the resistance of N-MP to heat and mass exchange that the same temperature conditions as for separation of DMAA—H_2O mixture are required for separation of N-MP—H_2O mixture. It follows from the temperature vs. N-MP partial pressure curve (Fig. 2) that the indicated temperature conditions are technically impossible to ensure in one column with the required number of separation steps. N-MP—H_2O mixture can only be totally separated in two separate columns — rectifying and stripping. The rectifying column is designed to remove the water contained in the solution; the stripping column is designed to obtain N-MP rectificate. The bottoms product from the rectifying column should be the feed for the stripping column, and the top product of the stripping column supplements the feed of the rectifying column. The target product should be sampled from the still part of the stripping column in the form of vapor phase. An absolute pressure of 25-50 kPa should then be maintained in the rectifying column still and a pressure of under 5 kPa should be maintained in the stripping column still.

These technological characteristics of vacuum rectification separation of N-MP—H_2O mixture can be used in planning industrial installations.

SEPARATION OF N-MP AND THE SALT COMPONENT OF A POLYMER SOLVENT BY CRYSTALLIZATION

The technological characteristics of this stage of regeneration are a function of the solvent and salt contained in the solution.

As in the general case, in the evolution of crystallization of a complex salt of N-MP with a salt component, it is necessary to have data on the character of the temperature curve of the solubility of the salt component in the solvent, the composition of the separated salt as a function of the temperature and composition of the solution, the effect of other impurities, etc.

A method which combines the methods of isothermal dissolution and isothermal crystallization was used in studying the temperature curve of the solubility of salts in N-MP. The maximum supersaturation obtained by the isothermal dissolution method was used for preparation of the initial solution and subsequent isothermal crystallization. This method allows reproducing the solubility results with simultaneous determination of the composition of the equilibrium solid phase. In addition, the required purity of both phases can be obtained when it is used (after separation of the suspension, there is no solid phase in the liquid and a minimum concentration of liquid phase in crystallites).

TABLE 1. Composition of Equilibrium Phases in Crystallization from Saturated Solutions Containing N-MP and a Salt Component

System	Crystallization temperature, °C	Fraction of salt component in equilibrium liquid phase, wt.%	Composition of equilibrium solid phase
$LiC\ell$ - N-MP	2	7,16	LiCl·N-MP solvate
	5	7,25	
	10	7,3	
	20	7,6	
	30	7,8	
	40	8,4	
	50	9,0	
	60	9,8	
	70	10,8	
	80	12,0	
$CaC\ell_2$ - N-MP	5	7,5	$CaCl_2$·3N-MP solvate
	10	7,7	
	20	7,8	
	30	8,1	
	40	8,3	
	50	8,8	
	55	8,9	
	60	9,4	
	70	10,2	
	75	10,6	

TABLE 2. Composition of Equilibrium Phases in Crystallization from Saturated Solutions Containing DMAA and a Salt Component

System	Crystallization temperature, °C	Fraction of salt component in equilibrium liquid phase, wt. %	Composition of equilibrium solid phase
$LiC\ell$ - DMAA	2,5	6,50	$LiC\ell$· DMAA
	5	6,80	
	10	7,00	
	20	7,60	
	40	9,20	
	50	9,60	
	60	10,50	
	70	11,10	
	80	11,90	
$CaC\ell_2$ - DMAA	5	1,68	$CaC\ell_2$·5 DMAA
	13	1,98	
	17	2,50	
	20	2,70	
	27	3,15	
	30	3,25	
	35	3,80	
	40	4,10	
$CaC\ell_2$ - DMAA	45	4,10	$CaC\ell_2$·3 DMAA
	50	3,93	
	55	3,75	
	60	3,65	
	65	3,58	
	70	3,55	
	75	3,55	

A saturated solution was prepared by dissolving the salt in the solvent at a temperature 10-15°C higher than the desired temperature while mixing constantly. The dissolved salt was then separated, and crystallization was conducted from the saturated solution while decreasing the temperature to the desired temperature. The liquid phase was periodically sampled through filtering packing. When a constant concentration of the salt component in the liquid phase was attained, crystallization ended.

Dissolution and crystallization were conducted in a hermetically sealed flask with a stirrer and jacket through which water circulated with the temperature set by a thermostat. A temperature 20°C lower was obtained by circulation of cold water and addition of ice. The suspension was separated on a LZ-401 laboratory centrifuge (Hungary) redesigned to filter with

TABLE 3. Phase Equilibrium in the LiCl—N-MP—H_2O in Crystallization (20°C)

Composition of initial solution, wt. %			Composition of equilibrium phases, wt. %					
			solid			liquid		
LiCl	N-MP	H_2O	LiCl	N-MП	H_2O	LiCl	N-MP	H_2O
10,0	89,7	0,3	27,40	72,50	0,10	7,57	92,08	0,35
10,0	89,2	0,8	26,90	72,70	0,40	7,85	91,03	1,12
11,4	87,1	1,5	24,80	74,50	0,70	8,44	89,73	1,83
11,4	86,6	2,0	26,80	72,26	0,94	9,20	88,40	2,40
11,5	85,5	3,0	23,98	74,19	1,83	9,80	86,70	3,50
12,3	84,1	3,6	21,60	68,40	9,99	9,92	86,25	3,83
12,3	84,2	3,5	22,15	67,35	10,50	9,60	86,68	3,72
10,0	85,0	5,0	23,00	64,36	12,64	9,02	85,57	5,41
10,6	81,55	7,85	22,90	65,80	11,30	9,50	82,10	8,40
10,3	81,4	8,3	19,80	68,90	11,30	9,67	80,36	9,97
10,5	80,1	9,4	22,50	57,10	20,40	10,00	79,70	10,30
10,0	76,4	13,6	21,40	57,30	21,30	8,10	77,90	14,00
10,0	74,0	16,0	20,30	56,70	23,00	9,08	73,72	17,20
11,0	69,0	20,0	21,35	57,58	21,07	10,10	68,20	21,70
12,5	62,5	25,0	17,40	56,70	25,90	11,80	61,00	27,20
14,0	56,0	30,0	–	–	–	13,24	56,86	29,90
16,0	49,0	35,0	–	–	–	14,90	48,70	36,50
18,0	42,0	40,0	–	–	–	17,10	43,40	39,50
22,0	34,0	44,0	22,30	52,40	25,20	20,10	35,20	44,80
22,0	33,0	45,0	22,50	52,30	25,20	20,50	33,20	46,30
25,0	27,0	48,0	22,70	47,30	30,00	24,20	27,10	48,70
30,0	19,0	51,0	23,10	49,10	27,80	28,10	20,30	51,60
41,2	14,8	44,0	61,50	3,70	34,80	36,40	11,40	52,20
43,8	4,7	51,5	62,90	3,40	33,70	40,65	6,45	52,90
46,5	0	53,5	69,50	0	30,50	43,50	0	56,50

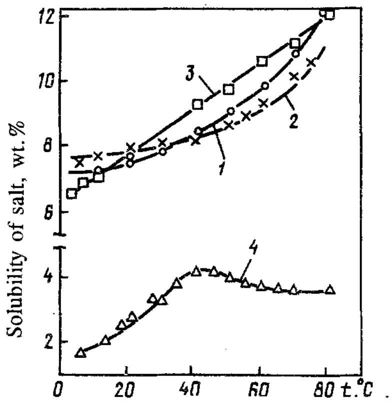

Fig. 3. Solubility of LiCl (1, 3) and $CaCl_2$ (2, 4) in N-MP (1, 2) and DMAA (3, 4) vs. temperature *t*.

special hermetically sealed filtering elements. The initial anhydrous components (salt and solvent) for obtaining a saturated solution were loaded and the suspension was unloaded from the crystallizers into the filtering elements and the separated equilibrium phases (crystals and liquid phase) were unloaded in a dry chamber in nitrogen medium.

LiCl—N-MP and $CaCl_2$—N-MP systems were investigated. The concentration of the salt component (LiCl, $CaCl_2$) was determined by the chlorine-ion method of potentiometric titration with mercury nitrate [13]. The results of the studies are reported in Table 1.

An increase in the solubility with an increase in the temperature was characteristic of both systems in the temperature range investigated (5-80°C). The absolute values of the solubility of LiCl and $CaCl_2$ in N-MP were very close, and the concentration of the equilibrium solid phase was constant. For a suspension separation factor of 500, the concentration of salt component in the complex salt formed by LiCl and N-MP was 24.5-25%, which corresponds to LiCl·N-MP monosolvate; in the complex salt formed by $CaCl_2$ and N-MP, the concentration of salt component was 23-25%, which corresponds to solvate of the composition $CaCl_2 \cdot 3$N-MP.

For comparison, the data for LiCl—DMAA and $CaCl_2$—DMAA systems obtained by a similar method are reported in the same temperature range in Table 2 and Fig. 3. As these data suggest, the behavior of both solvents (N-MP and DMAA)

TABLE 4. Phase Equilibrium in the LiCl—DMAA—H_2O System during Crystallization (20°C)

Composition of starting solution, wt. %			Composition of equilibrium phases, wt. %					
			solid			liquid		
LiCl	DMAA	H_2O	LiCl	DMAA	H_2O	LiCl	DMAA	H_2O
10,20	89,60	0,32	29,50	70,36	0,14	8,30	90,92	0,78
10,78	88,65	0,67	29,50	70,14	0,28	8,50	90,23	1,27
10,70	88,20	1,33	30,00	69,40	0,60	9,02	88,82	2,16
11,40	86,30	2,60	29,50	69,87	0,72	9,53	87,36	3,10
11,00	85,10	3,90	29,42	68,00	2,60	10,65	84,65	4,70
11,40	83,54	5,30	29,30	67,55	3,15	10,80	83,30	5,89
12,07	81,75	6,25	26,60	68,00	5,40	11,00	82,38	6,62
12,60	80,10	7,90	23,94	55,86	20,20	11,88	79,30	8,83
12,30	78,90	8,80	23,20	53,40	23,40	10,80	78,40	10,80
11,80	77,60	10,60	22,91	52,60	24,50	10,40	76,20	13,40
13,30	72,90	13,80	23,86	52,64	23,50	10,85	70,80	18,35
13,30	71,10	15,60	23,20	50,10	26,70	10,70	69,70	19,60
13,30	69,30	17,40	24,64	50,16	25,20	10,60	68,60	20,80
15,70	64,65	19,65	22,56	53,74	23,70	11,73	65,37	22,90
15,70	63,23	21,07	22,18	53,86	23,96	12,40	62,74	24,86
14,77	62,52	22,72	23,12	50,58	26,30	12,42	62,38	25,20
14,28	60,02	25,70	22,48	53,95	23,57	13,10	57,80	39,10
16,70	54,10	29,20	22,40	53,50	24,10	14,00	56,00	30,00
18,90	51,40	29,70	21,30	55,90	22,80	15,47	54,13	39,40
23,00	46,20	30,80	25,90	50,20	23,90	18,30	45,20	36,50
21,90	43,10	35,00	24,20	43,70	26,10	19,20	41,50	39,30
21,10	42,00	36,90	24,40	51,70	23,90	20,60	37,50	41,90
25,00	37,50	37,50	25,96	48,07	25,97	23,10	30,90	48,00
25,40	33,80	40,80	24,00	51,33	24,67	22,00	34,50	43,50
22,30	37,20	41,50	24,07	52,43	23,50	23,00	32,20	44,80
35,60	12,90	51,50	29,50	44,40	26,10	34,30	13,50	52,20
42,40	7,55	50,00	65,30	6,58	28,12	39,08	8,28	52,64
43,40	3,80	53,77	65,20	38,00	31,00	40,30	5,70	54,00
44,85	3,77	52,40	67,84	1,38	30,78	43,30	3,20	53,50
45,45	0	54,54	69,50	0	30,50	43,50	0	56,50

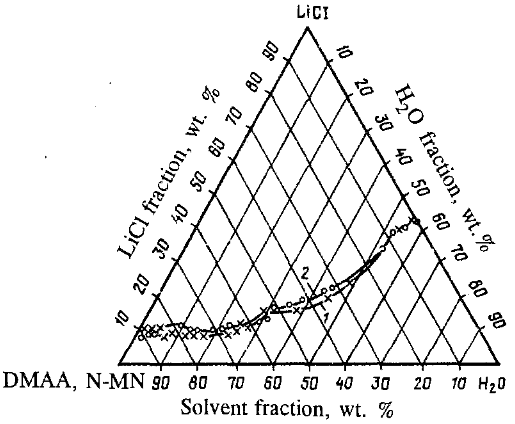

Fig. 4. Isotherm of the solubility of LiCl in N-MP—H_2O (1) and DMAA—H_2O systems (2) at 20°C.

with respect to LiCl is almost the same; the absolute values of the solubility are very close in the entire temperature range. These solvents behave differently with respect to $CaCl_2$: in the temperature range investigated, the solubility of $CaCl_2$ in DMAA is much lower than in N-MP; in addition, in contrast to the $CaCl_2$—N-MP system, the solubility of $CaCl_2$ only increases in the $CaCl_2$—DMAA system below the temperature of 40°C, and the concentration of $CaCl_2$ decreases from 4.1 to 3.55% up to 75°C; the equilibrium solid phase below 40°C is $CaCl_2 \cdot 5DMAA$ solvate, while it is $CaCl_2 \cdot 3DMAA$ solvate above 40°C.

The shape and size of the crystals were evaluated under a microscope in an immersion liquid, since the products are hygroscopic. PMS-6 polymethylsiloxane was used as the immersion liquid. The shape of the crystals of the LiCl complex with the solvent is similar to $LiCl \cdot H_2O$ monohydrate, and the shape of crystals of the $CaCl_2$ complex with the solvent is similar to $CaCl_2$ hydrates. The analysis of the separated crystals also showed that supersaturation (the concentration of salt in the saturated solution should be 2-3% higher) is required and a sharp reduction in the temperature and intense stirring are inadmissible for obtaining relatively large crystals (>150 μm) during crystallization.

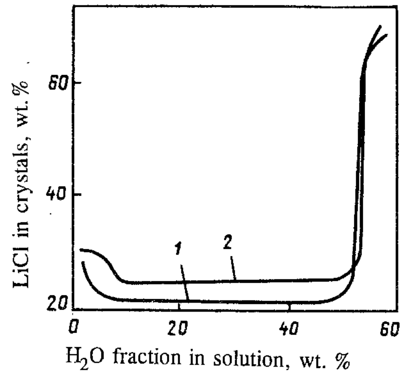

Fig. 5. Concentration of LiCl in crystals of the complex salt vs. composition of the solution: 1) N-MP—H_2O; 2) DMAA—H_2O.

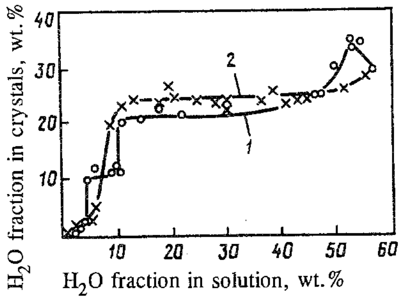

Fig. 6. Concentration of H_2O in crystals of the complex salt vs. composition of the solution: 1) N-MP—H_2O; 2) DMAA—H_2O.

Crystals measuring 5 mm and larger can be obtained with natural cooling of a saturated solution for 1-2 days without stirring.

The LiCl—N-MP—H_2O system was studied at the same time as the LiCl—N-MP system. The composition of the equilibrium phases during crystallization was investigated as a function of the concentration of water in the initial solution, which allowed formulating the requirements for the composition of the initial solution for crystallization of the LiCl·N-MP complex salt containing almost no water impurity. The results of the studies are reported in Table 3.

As for the simpler system (salt—solvent), the solubility of LiCl in the LiCl—N-MP—H_2O system was investigated with two methods: isothermal dissolution and subsequent crystallization. The difference was that seed crystals separated from a similar solution at 5-7°C were added to the mixture of solvent and water during crystallization due to the ability of these solutions to create strong supersaturation during dissolution of the salt in the mixture. The concentration of salt component and water was determined in the crystals and equilibrium solution. The concentration of water was determined by titration with Fischer's reagent. For comparison, the LiCl—DMAA—H_2O system was investigated in identical conditions. The results are reported in Table 4.

As Figs. 4-6 show, salts of different composition are separated when the concentration of water in the solution is changed. Six branches, whose points of intersection indicate a qualitative change in the equilibrium solid phase, are characteristic of equilibrium in the LiCl—DMAA—H_2O system. The separate branches reflect a continuous change in the properties of the system as a function of the composition.

The basic salts separated are: LiCl·N-MP (30+70%), LiCl·N-MP·H_2O (26.7+62+11.3%), LiCl·N-MP·$2H_2O$ (23.8+55.8+20.4), LiCl·H_2O (70+30%). In pure form, the LiCl·N-MP salt can only be separated in a concentration of less than 0.1% in the initial solution. With a higher concentration of water — up to 3.5%, LiCl·N-MP·H_2O crystals are separated; LiCl·N-MP·H_2O crystals are separated from 3.5 to 10% H_2O;LiCl·N-MP·$2H_2O$ crystals are separated with 10 to 40-45% H_2O;

with a concentration of H_2O greater than 45%, the equilibrium composition of the crystals is represented by a mixture of $LiCl \cdot N\text{-}MP \cdot 2H_2O$ and $LiCl \cdot H_2O$ salts.

The LiCl—DMAA—H_2O system is characterized by four branches, and the basic salts separated are: LiCl·DMAA (33+67%), $LiCl \cdot DMAA \cdot 2H_2O$ (25.6+52.6+21.8%), $LiCl \cdot H_2O$ (70+30%).

In pure form, LiCl·DMAA salt, like LiCl·N-MP, can only be separated with an approximately 0.1% concentration of water; the equilibrium composition of the crystals is represented by a mixture of LiCl·DMAA and $LiCl \cdot DMAA \cdot 2H_2O$ salts with a higher concentration of water, up to 8%; $LiCl \cdot DMAA \cdot 2H_2O$ salt for 8 to 52% water, and a mixture of $LiCl \cdot DMAA \cdot 2H_2O$ and $LiCl \cdot H_2O$ salts above 52%.

As these data suggest, the differences in the LiCl—N-MP—H_2O and LiCl—DMAA—H_2O systems are not major. For this reason, the industrial experience in separation of LiCl·DMAA complex salt from spent technological solutions and preparation of a solvent composite based on it can be used for separation of the salt component from technological solutions containing N-MP.

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