

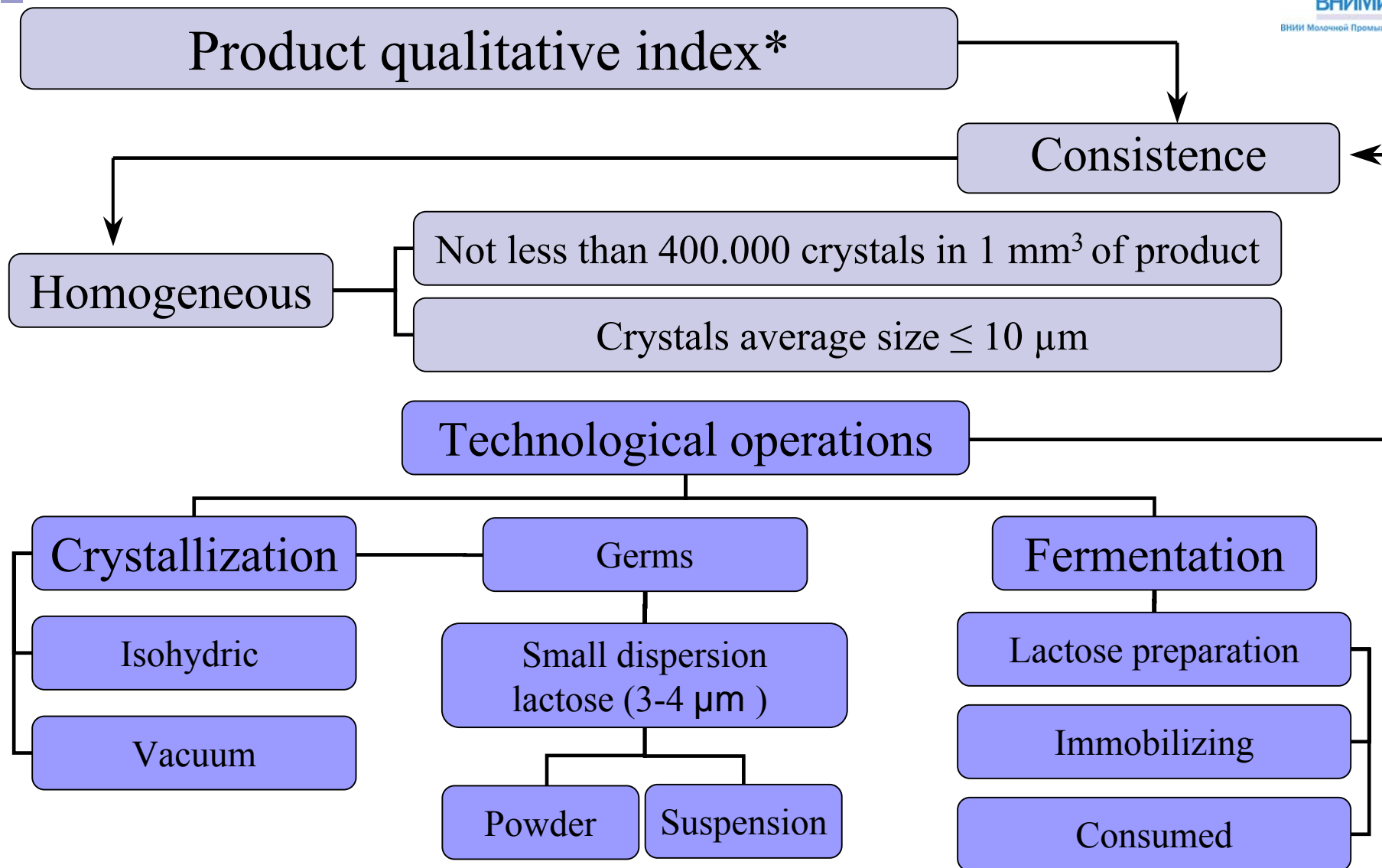


ВНИИ Молочной Промышленности

# LACTOSE CRYSTALLIZATION FROM SATURATED SOLUTIONS

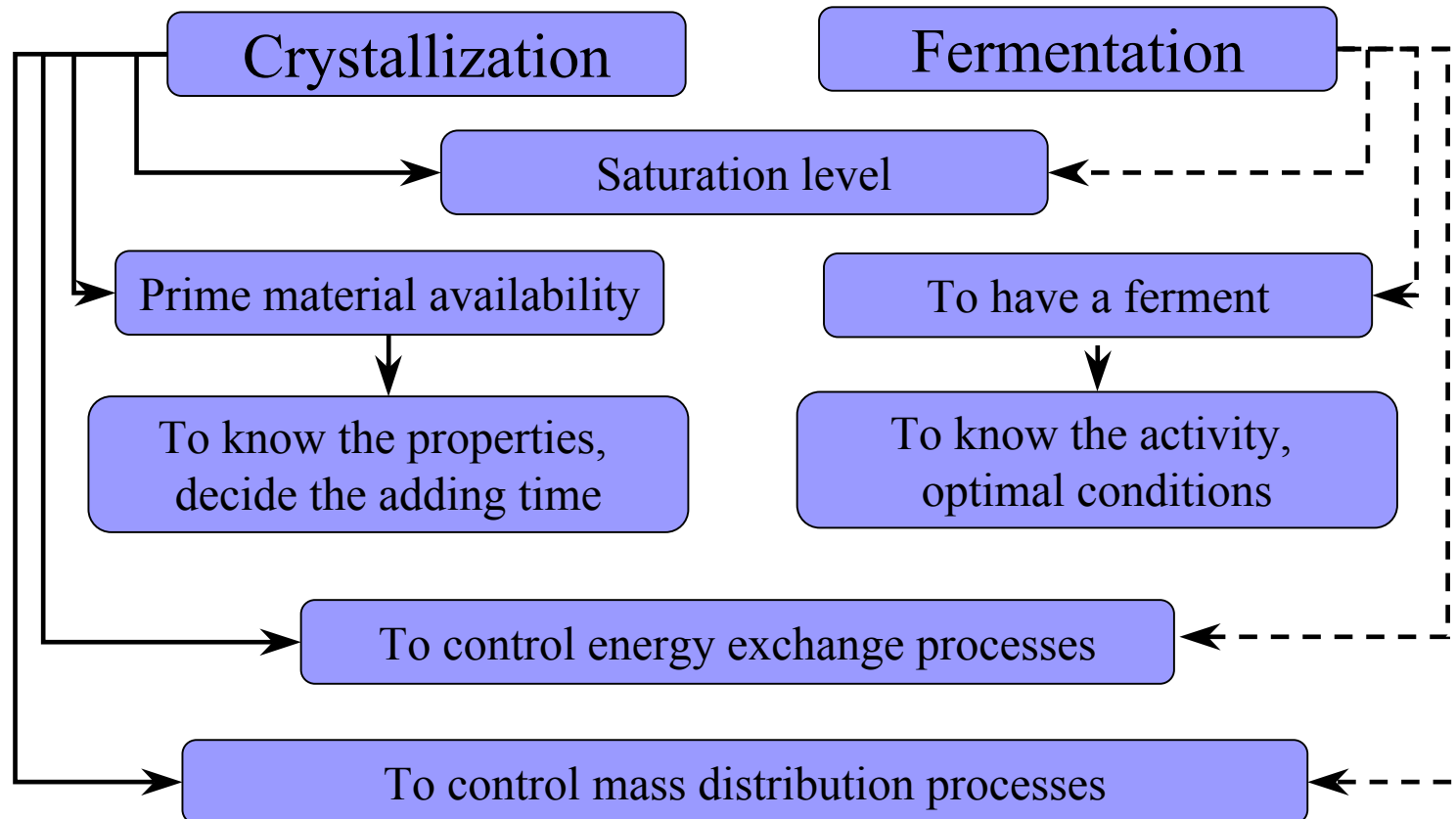
Aram Galstyan

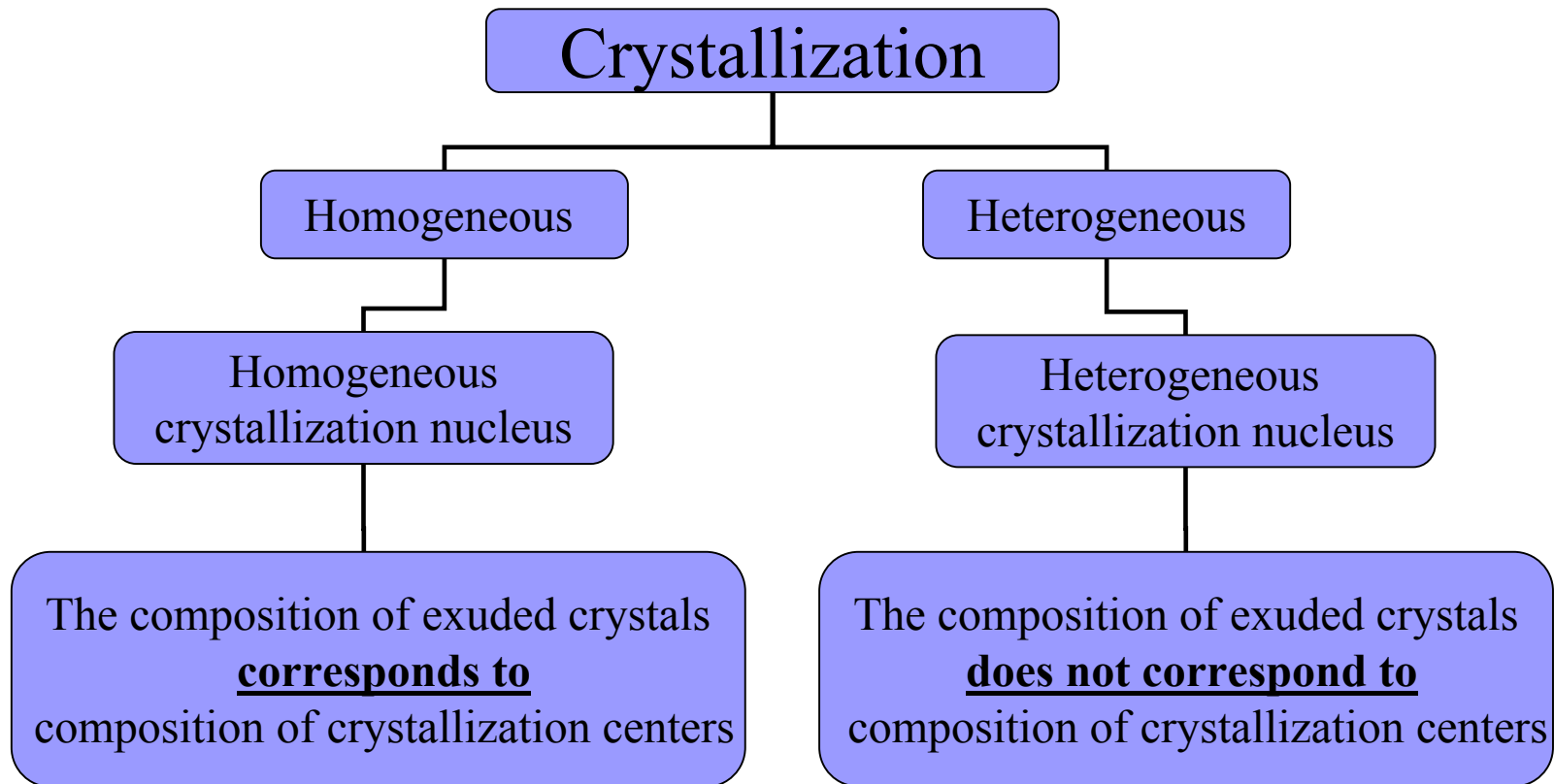
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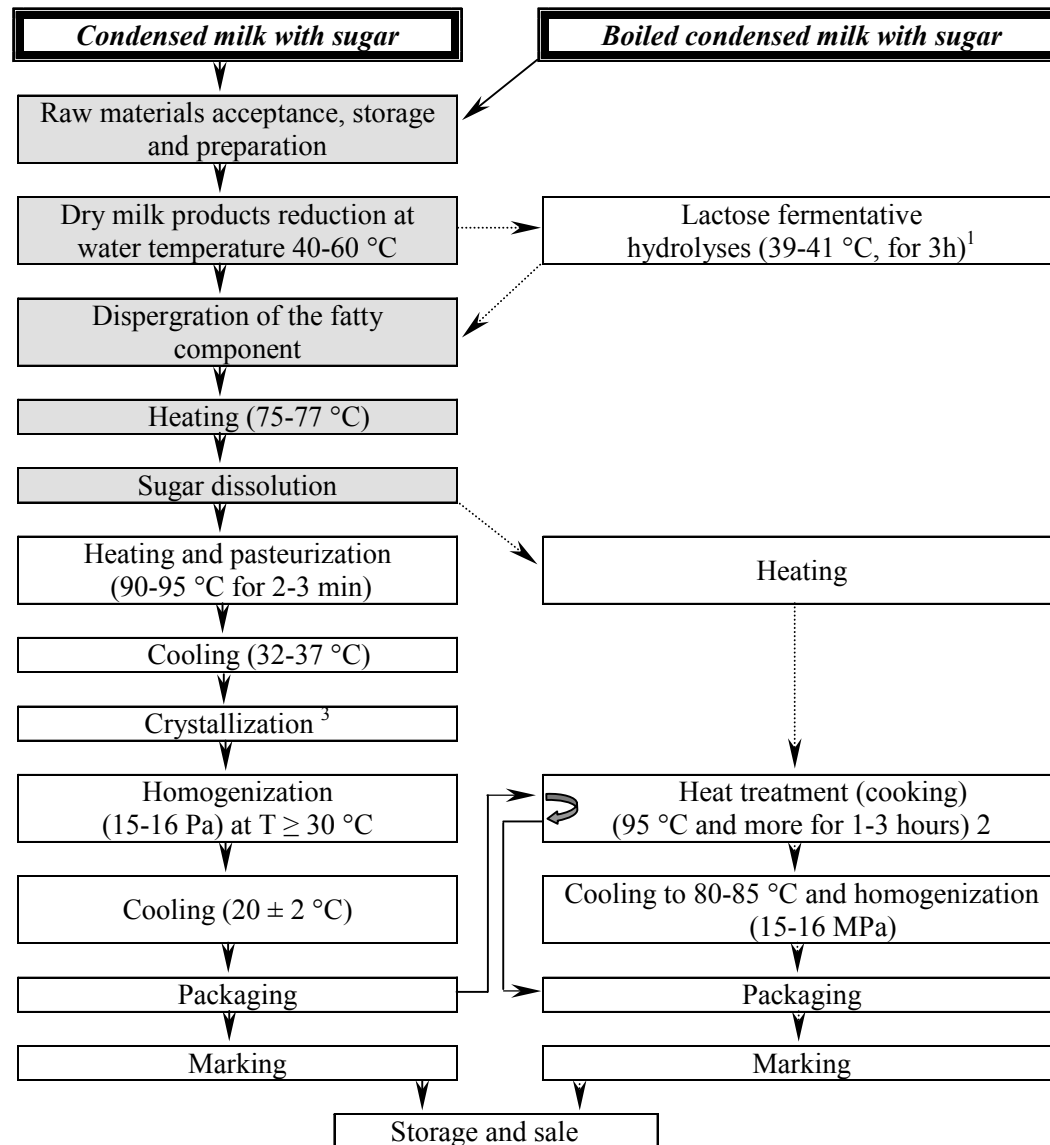
# *Technological requirements*





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# Principle technological schemes of manufacture of sugar containing condensed milk products



<sup>1</sup> Employed with a purpose of prevention the uncontrolled crystallization & speeding-up cooking time

<sup>2</sup> Duration depends on the temperature & fermentation process

<sup>3</sup> The adding of priming material in vacuum meant. In manufacture of sugar-containing condensed milk “cooking” is not employed.

## Model product's normalized indices

<b>Index appellation</b>	<b>Standards</b>
Mass proportion of moisture, %	$\leq 26,5$
Mass proportion of saccharose, %	$\geq 43,5$
Total mass proportion of milk dry materials, % <i>also fat, %</i>	$\geq 28,5$ $\geq 8,5$
Acidity, °T	$\leq 45$
Viscosity, Pa*c <i>valid within 2 months</i> <i>valid within 2 to 12 months</i>	$\leq 3-6$ 15
Allowed size of lactose crystals, $\mu\text{m}$ ,	$\leq 15$

# Recipes of sugar-containing condensed milk products and established conventional coding

Components	Recipes	Coding
Dry skimmed milk (dry materials proportion 95%, fat proportion 1,5%)	213,0	$X_1$
Milk fat (fat proportion 99,8%)	82,0	$X_2$
Granulated sugar (dry materials proportion 99,8%)	450,0	$X_3$
Water	255,0	$X_4$
Total product	1000,0	$X_5$

Appellation of the operation	Coding	Appellation of the operation	Coding
<i>Dissolution</i>	$Y_1$	<i>Vacuuming</i>	$Y_8$
<i>Germs bringing in</i>	$Y_2$	<i>Crystallization</i>	$Y_9$
<i>Dispergration</i>	$Y_3$	<i>Cooking</i>	$Y_{10}$
<i>Heating</i>	$Y_4$	<i>Packing</i>	$Y_{11}$
<i>Homogenization</i>	$Y_5$	<i>Mixing</i>	$Y_{12}$
<i>Pasteurization</i>	$Y_6$	<i>Marking</i>	$Y_{13}$
<i>Cooling</i>	$Y_7$	<i>Storing</i>	$Y_{14}$

↓

$$X_5 = X_4 + X_1 + X_2 + X_3$$

## Operative models: condensed milk with sugar (A,B,C) and condensed milk with sugar “cooked” (C')

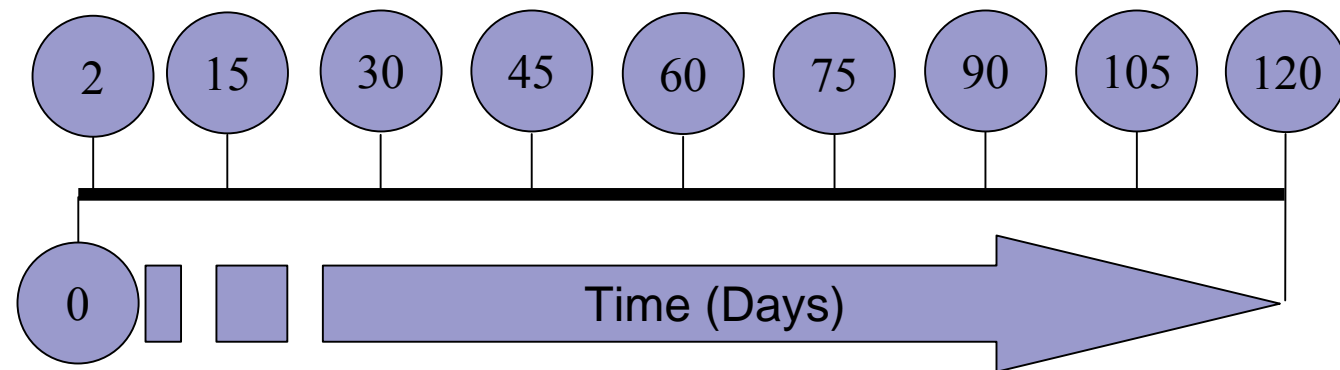
Operations sequence	<i>Possible modification of germs adding time</i>			
	<b>A</b>	<b>B</b>	<b>C</b>	<b>C'</b>
TO1	$Y_4 X_4 \leftarrow Z$	$Y_4 X_4$	$Y_4 X_4$	$Y_4 X_4$
TO2	$Y_1 X_1$	$Y_1 X_1$	$Y_1 X_1$	$Y_1 X_1$
TO3	$Y_4 X_{41}$	$Y_4 X_{41}$	$Y_4 X_{41}$	$Y_4 X_{41}$
TO4	$Y_3 X_2$	$Y_3 X_2$	$Y_3 X_2$	$Y_3 X_2$
TO5	$Y_5 X_{412}$	$Y_5 X_{412}$	$Y_5 X_{412}$	$Y_5 X_{412}$
TO6	$Y_4 X_{412}$	$Y_4 X_{412} \leftarrow Z$	$Y_4 X_{412}$	$Y_4 X_{412}$
TO7	$Y_1 X_3$	$Y_1 X_3$	$Y_1 X_3$	$Y_1 X_3$
TO8	$Y_6 X_{4123}$	$Y_6 X_{4123}$	$Y_6 X_{4123}$	$Y_6 X_{4123}$
TO9	$Y_8 X_{4123}$	$Y_8 X_{4123}$	$Y_8 X_{4123} \leftarrow Z$	$Y_8 X_{4123} \leftarrow Z$
TO10	$Y_7 X_{4123}$	$Y_7 X_{4123}$	$Y_7 X_{4123}$	$Y_7 X_{4123}$
TO11	$Y_9 X_{4123}$	$Y_9 X_{4123}$	$Y_9 X_{4123}$	$Y_{11} X_5$
TO12	$Y_{11} X_5$	$Y_{11} X_5$	$Y_{11} X_5$	$Y_{10} X_5$
TO13	$Y_{13} X_5$	$Y_{13} X_5$	$Y_{13} X_5$	$Y_7 X_5$
TO14	$Y_{14} X_5$	$Y_{14} X_5$	$Y_{14} X_5$	$Y_{13} X_5$
TO15	-	-	-	$Y_{15} X_5$

*TO* – technological operation; *Z* – time point of bringing in of germs



# Time point of sampling for microscoping

Storage  
temperature  
6-10<sup>0</sup>C



0 – end of technological process and beginning of analysis  
120 – end of analysis



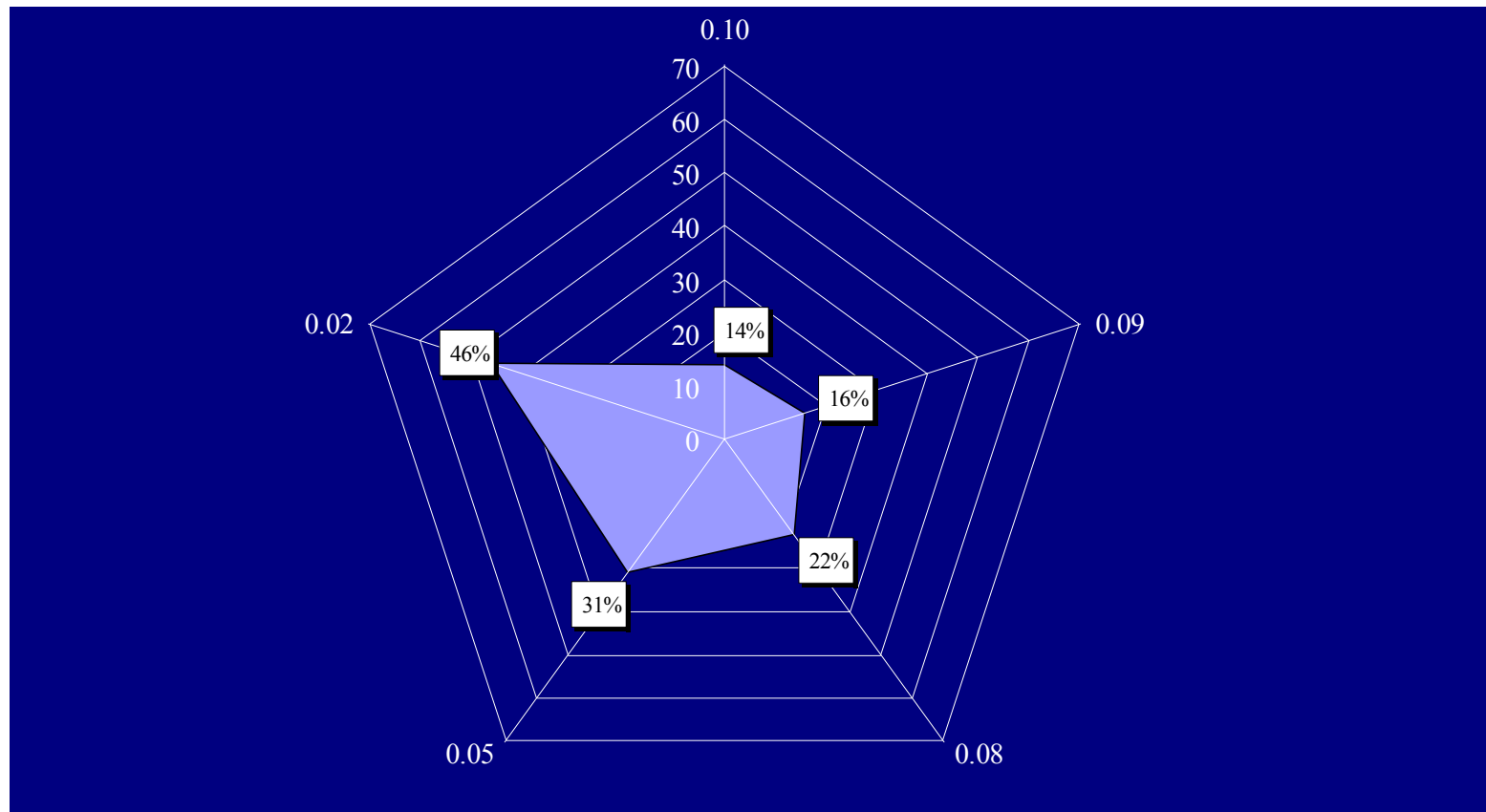
№	Products	Molecular formula	Properties, %	Solubility in water at pH≤7,0, mg/l
1	Silicon dioxide	SiO <sub>2</sub>	≥ 98	Not present
2	Titanium dioxide	TiO <sub>2</sub>		not present
3	Calcium carbonate	CaCO <sub>3</sub>		14-15

Crystals average size was calculated by formula: $D = \sum na / \sum n$	Coefficient of crystals homogeneity was calculated by the means of N. Figurovskii's formula $U = \Delta a \sqrt{\sum n / (2 \sum nv^2)}$
<i>D</i> – mean value of crystals size, μm ; <i>n</i> – crystals frequency; <i>a</i> – crystals linear size, μm	<i>U</i> – homogeneity coefficient; $\Delta a$ – crystal size limit, μm; <i>n</i> – crystals frequency; <i>v</i> – given and mean sizes divergence, μm

Crystals average size, μm	Lactose crystals likely amount in 1mm <sup>3</sup> of product, M	Crystals average size, μm	Lactose crystals likely amount in 1mm <sup>3</sup> of product, M
6	770 000	12	98 000
7	500 000	15	50 000
8	270 000	20	21 000
9	220 000	30	7 000
10	175 000	40	2 600

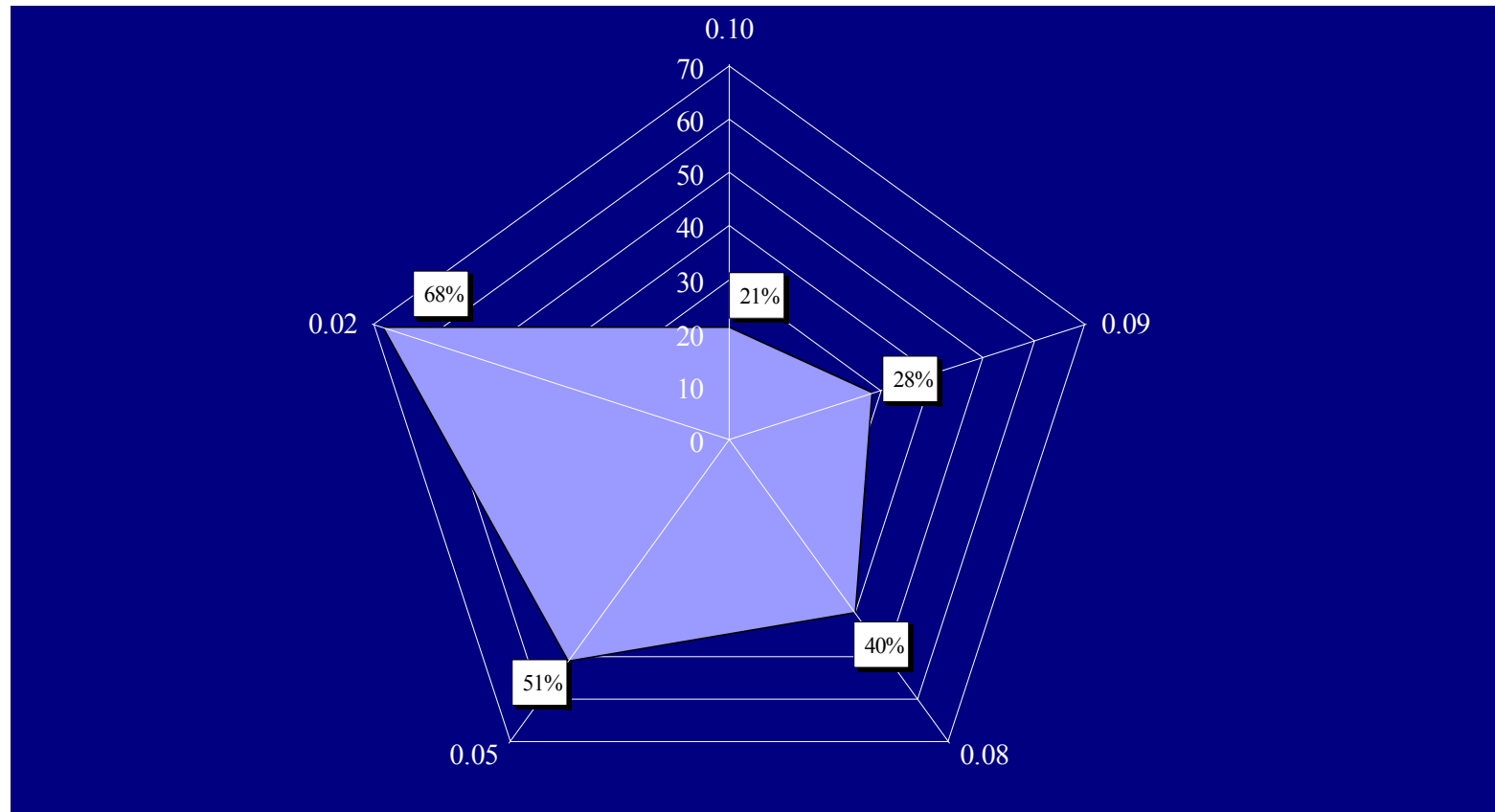
Distribution of samples with no crystallization effect when germ size is 3  $\mu\text{m}$  and its corresponding dosage 0,02...0,10% of product mass

Model C
$Y_4 X_4$
$Y_1 X_1$
$Y_4 X_{41}$
$Y_3 X_2$
$Y_5 X_{412}$
$Y_4 X_{412}$
$Y_1 X_3$
$Y_6 X_{4123}$
<b><math>Y_8 X_{4123} \leftarrow Z</math></b>
$Y_7 X_{4123}$
$Y_9 X_{4123}$
$Y_{11} X_5$
$Y_{13} X_5$
$Y_{14} X_5$



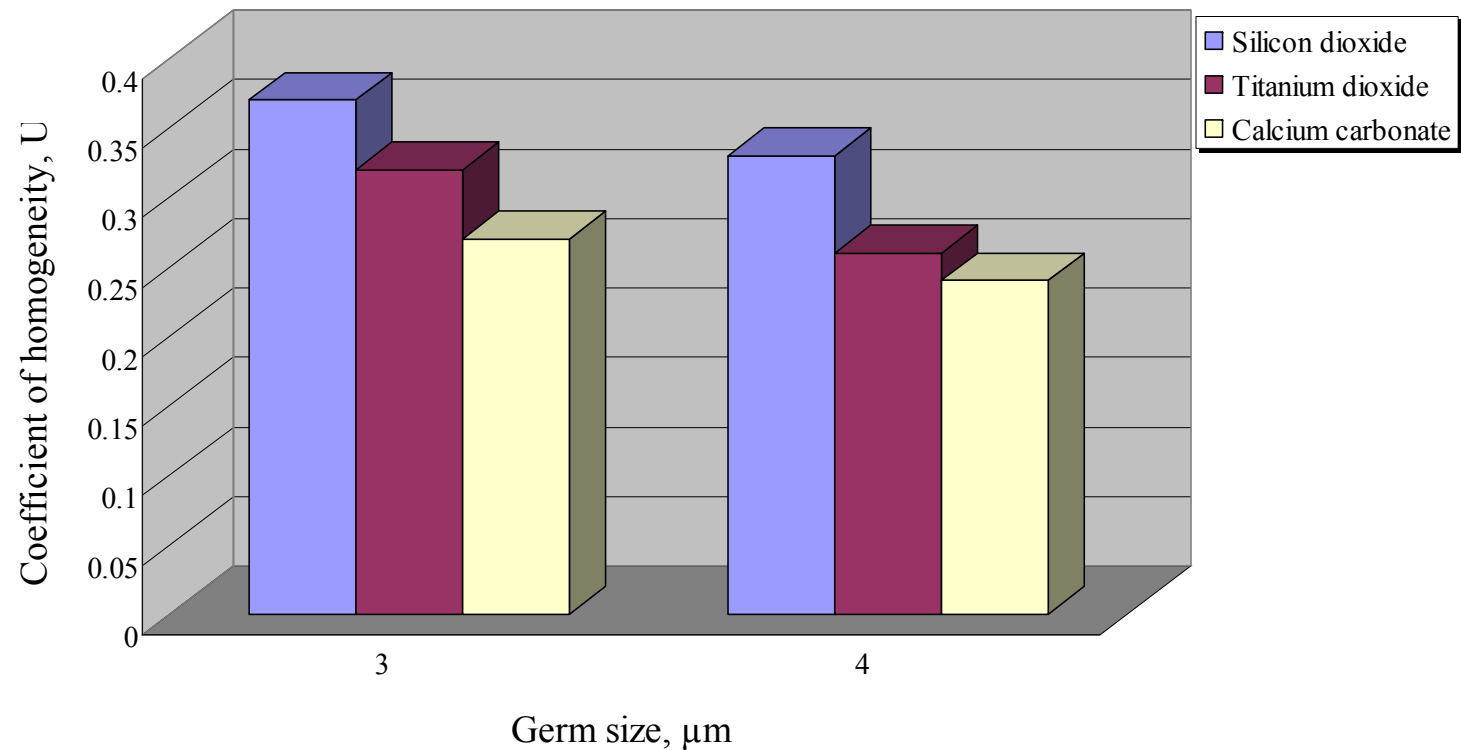
Distribution of samples with no crystallization effect when germ size is 4 μm and its corresponding dosage 0,02...0,10% of product mass

Model C
$Y_4 X_4$
$Y_1 X_1$
$Y_4 X_{41}$
$Y_3 X_2$
$Y_5 X_{412}$
$Y_4 X_{412}$
$Y_1 X_3$
$Y_6 X_{4123}$
<b><math>Y_8 X_{4123} \leftarrow Z</math></b>
$Y_7 X_{4123}$
$Y_9 X_{4123}$
$Y_{11} X_5$
$Y_{13} X_5$
$Y_{14} X_5$



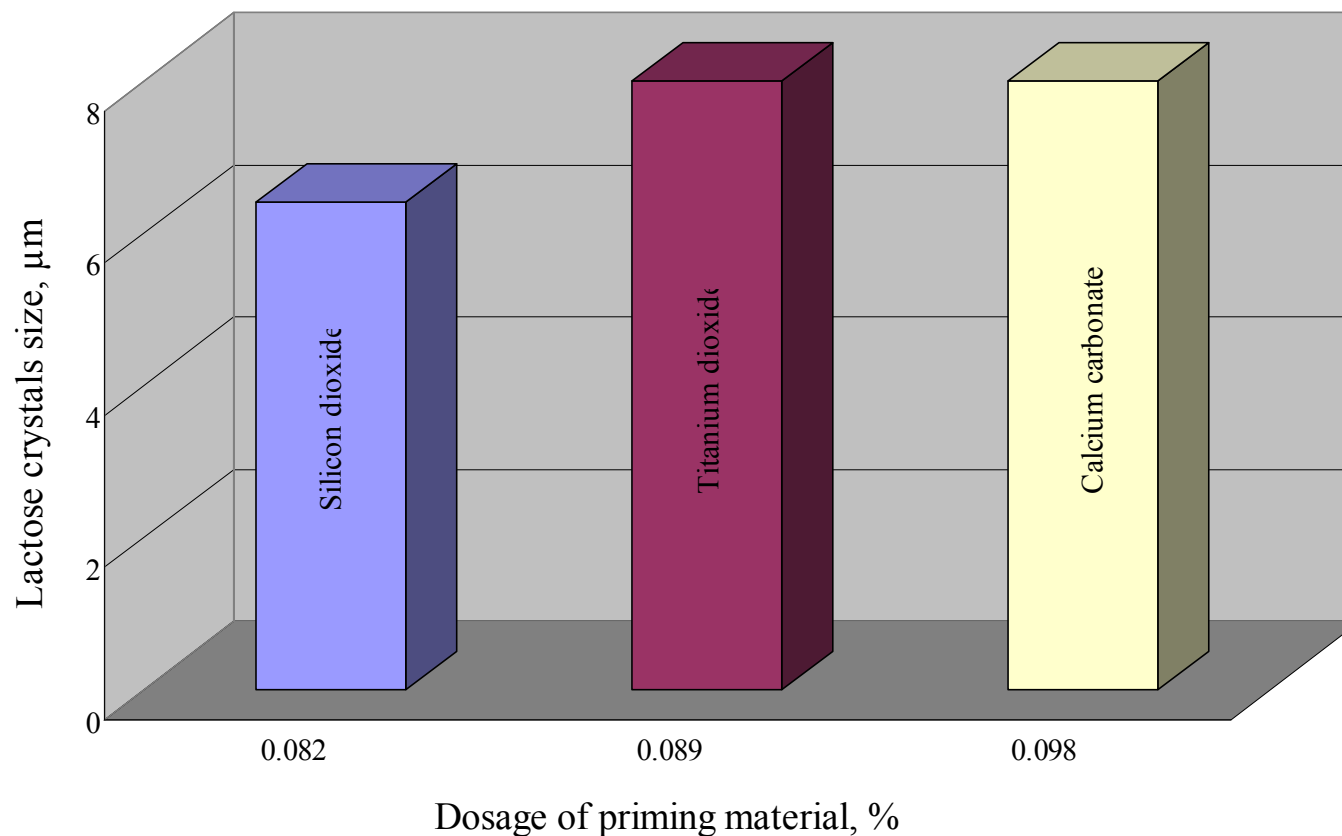
## Lactose crystals homogeneity coefficient when priming material size is 3-4 $\mu\text{m}$

Model C
$Y_4 X_4$
$Y_1 X_1$
$Y_4 X_{41}$
$Y_3 X_2$
$Y_5 X_{412}$
$Y_4 X_{412}$
$Y_1 X_3$
$Y_6 X_{4123}$
$Y_8 X_{4123} \leftarrow \mathbf{Z}$
$Y_7 X_{4123}$
$Y_9 X_{4123}$
$Y_{11} X_5$
$Y_{13} X_5$
$Y_{14} X_5$



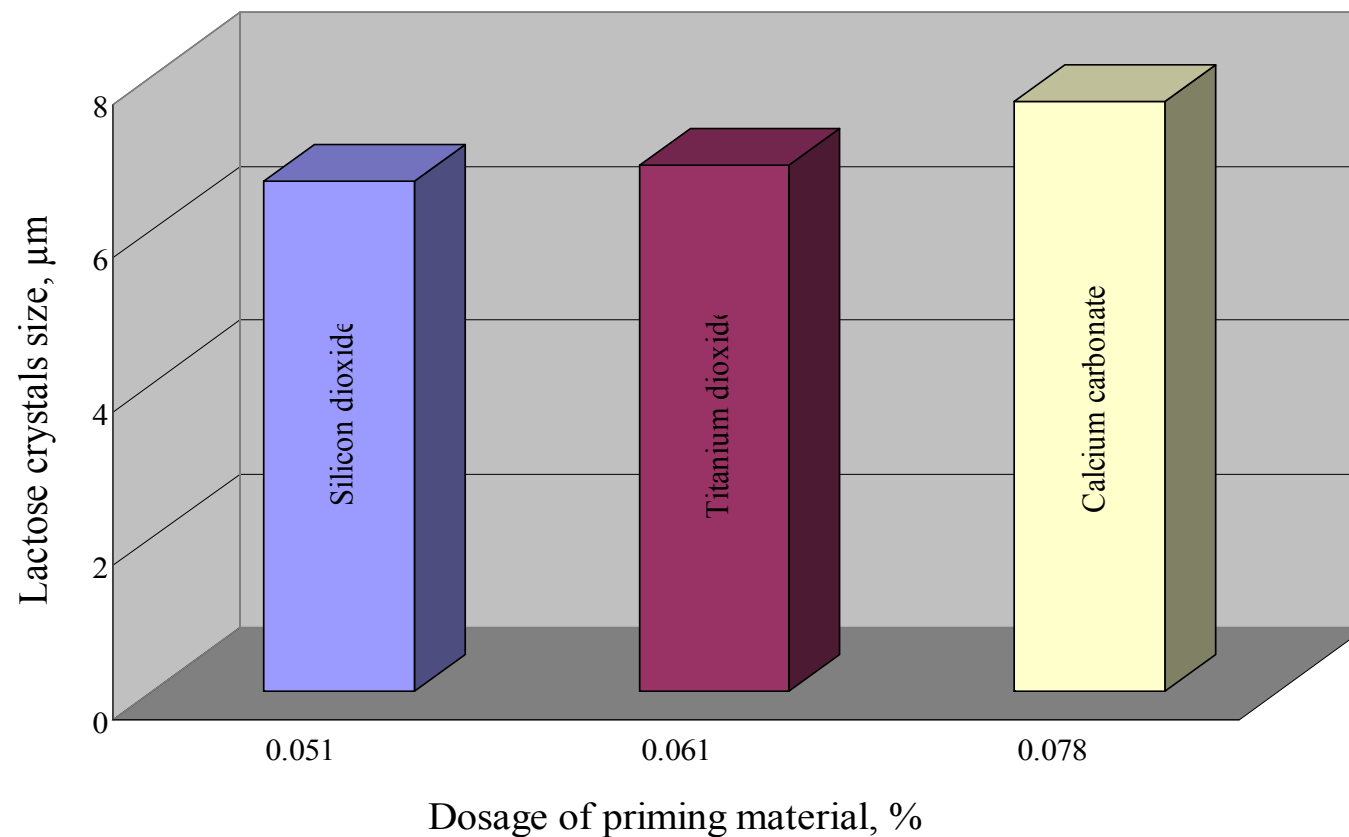
# Rational dosage of priming material for lactose crystallization by model A

Model A
$Y_4 X_4 \leftarrow Z$
$Y_1 X_1$
$Y_4 X_{41}$
$Y_3 X_2$
$Y_5 X_{412}$
$Y_4 X_{412}$
$Y_1 X_3$
$Y_6 X_{4123}$
$Y_8 X_{4123}$
$Y_7 X_{4123}$
$Y_9 X_{4123}$
$Y_{11} X_5$
$Y_{13} X_5$
$Y_{14} X_5$



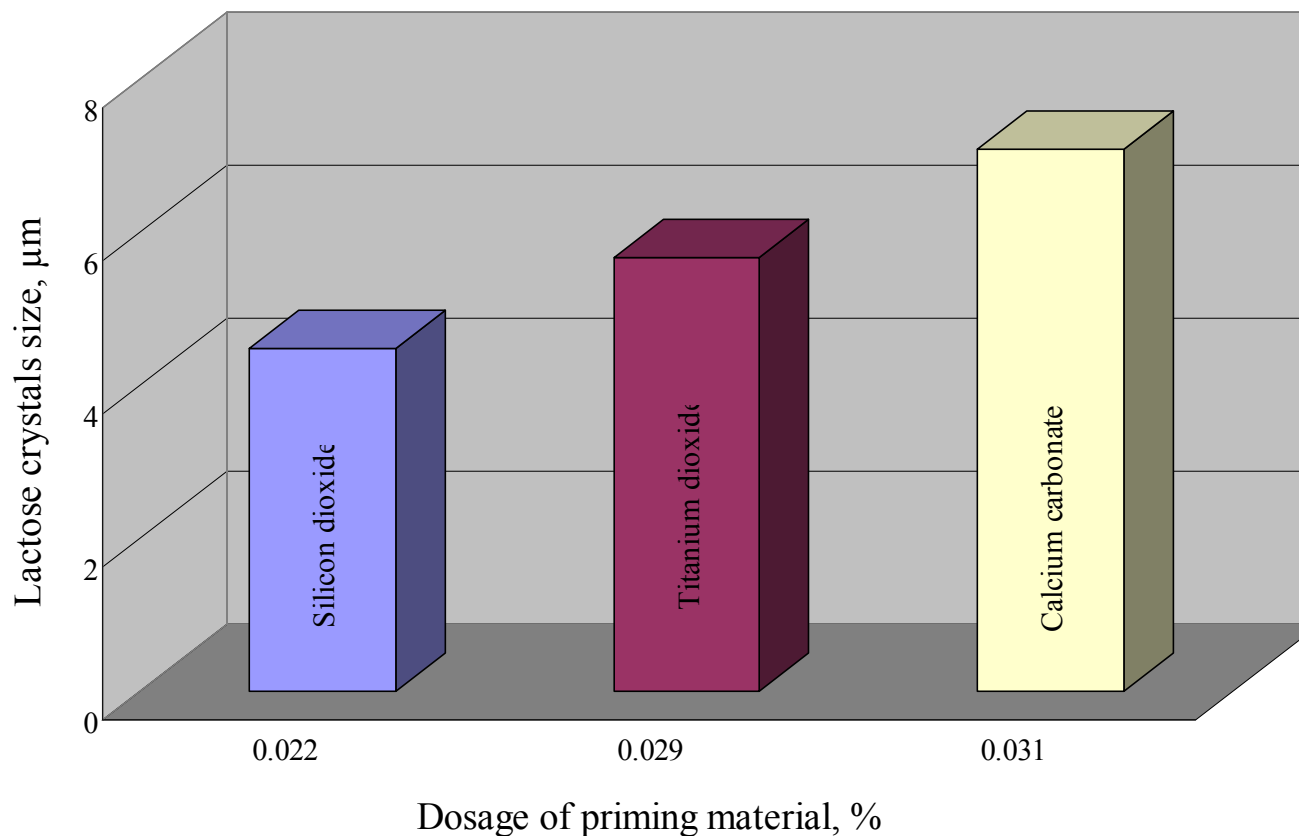
# Rational dosage of priming material for lactose crystallization by model B

Model B
$Y_4 X_4$
$Y_1 X_1$
$Y_4 X_{41}$
$Y_3 X_2$
$Y_5 X_{412}$
$Y_4 X_{412} \leftarrow Z$
$Y_1 X_3$
$Y_6 X_{4123}$
$Y_8 X_{4123}$
$Y_7 X_{4123}$
$Y_9 X_{4123}$
$Y_{11} X_5$
$Y_{13} X_5$
$Y_{14} X_5$



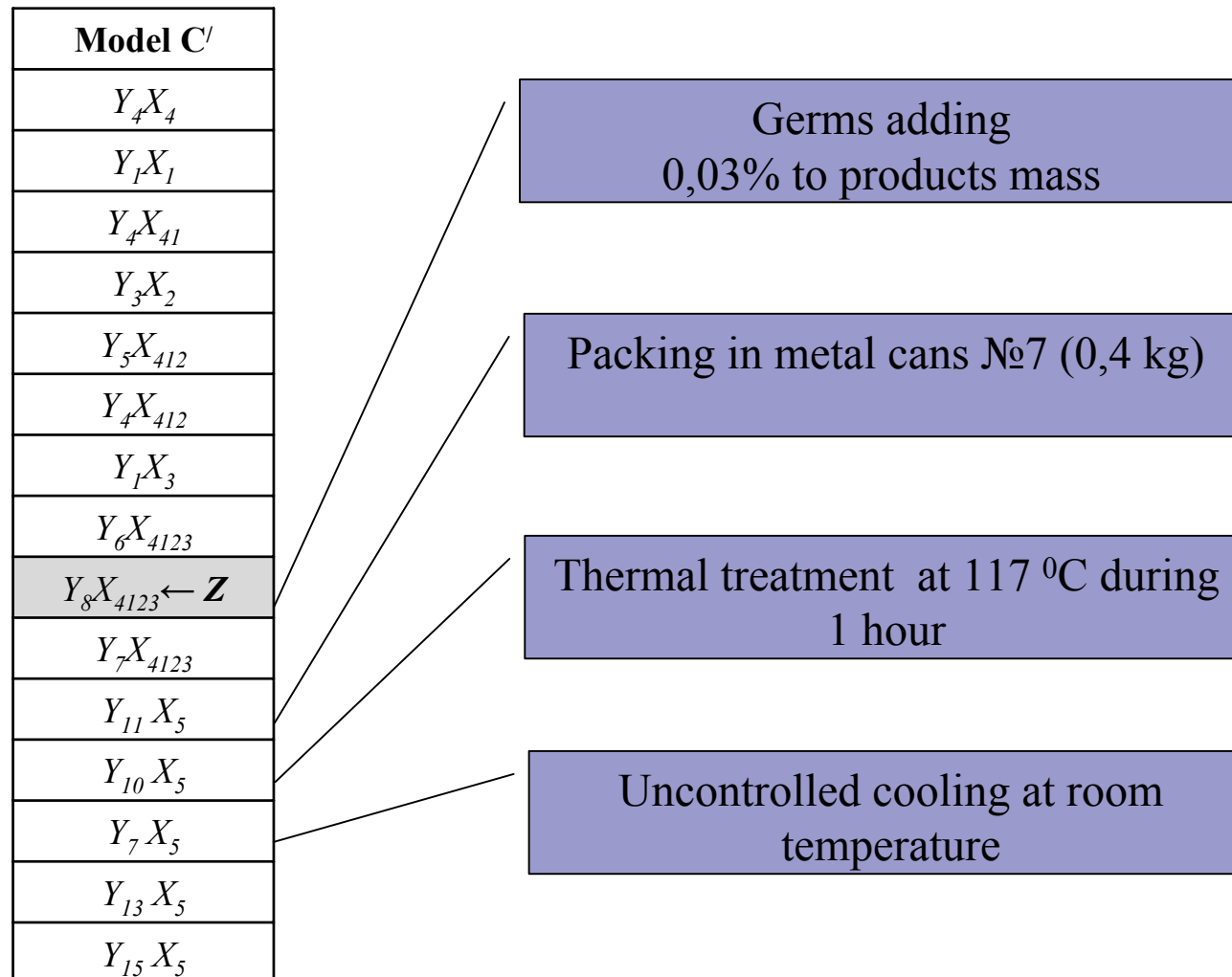
# Rational dosage of priming material for lactose crystallization by model C

Model C
$Y_4 X_4$
$Y_1 X_1$
$Y_4 X_{41}$
$Y_3 X_2$
$Y_5 X_{412}$
$Y_4 X_{412}$
$Y_1 X_3$
$Y_6 X_{4123}$
$Y_8 X_{4123} \leftarrow Z$
$Y_7 X_{4123}$
$Y_9 X_{4123}$
$Y_{11} X_5$
$Y_{13} X_5$
$Y_{14} X_5$





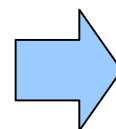
## Operative model of packaged product lactose crystallization after its thermal treatment



## Results of packaged product lactose crystallization after its thermal treatment

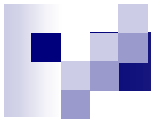
Model	Crystals group	Crystals size, $a$ , $\mu\text{m}$	Crystals frequency, $n$	Product $na$	Discrepancy, $\nu$	$\nu^2$	$n\nu^2$
$C'$	I	4	26	104	2.98	8.88	230.9
$Y_4 X_4$		6	14	84	0.98	0.96	13.5
$Y_1 X_1$		8	51	408	1.02	1.04	53.1
$Y_4 X_{41}$		10	4	40	3.02	9.12	36.5
$Y_3 X_2$	II	12	4	48	5.02	25.20	100.8
$Y_5 X_{412}$		14	1	14	7.02	49.28	49.3
$Y_4 X_{412}$	III	16	0	-	-	-	-
$Y_1 X_3$		18	0	-	-	-	-
$Y_6 X_{4123}$		20	0	-	-	-	-
$Y_8 X_{4123} \leftarrow Z$		22	0	-	-	-	-
$Y_7 X_{4123}$		24	0	-	-	-	-
$Y_{11} X_5$	IV	$\geq 25$	0	-	-	-	-
$Y_{10} X_5$							
$Y_7 X_5$		$\Delta a = 2$	$\sum n = 100$	$\sum an = 698$	-	-	$\sum n\nu^2 = 484.1$
$Y_{13} X_5$							
$Y_{15} X_5$		$M = \sum na / \sum n = 6.98$			$U = 0.64$		

Efficacy gradation

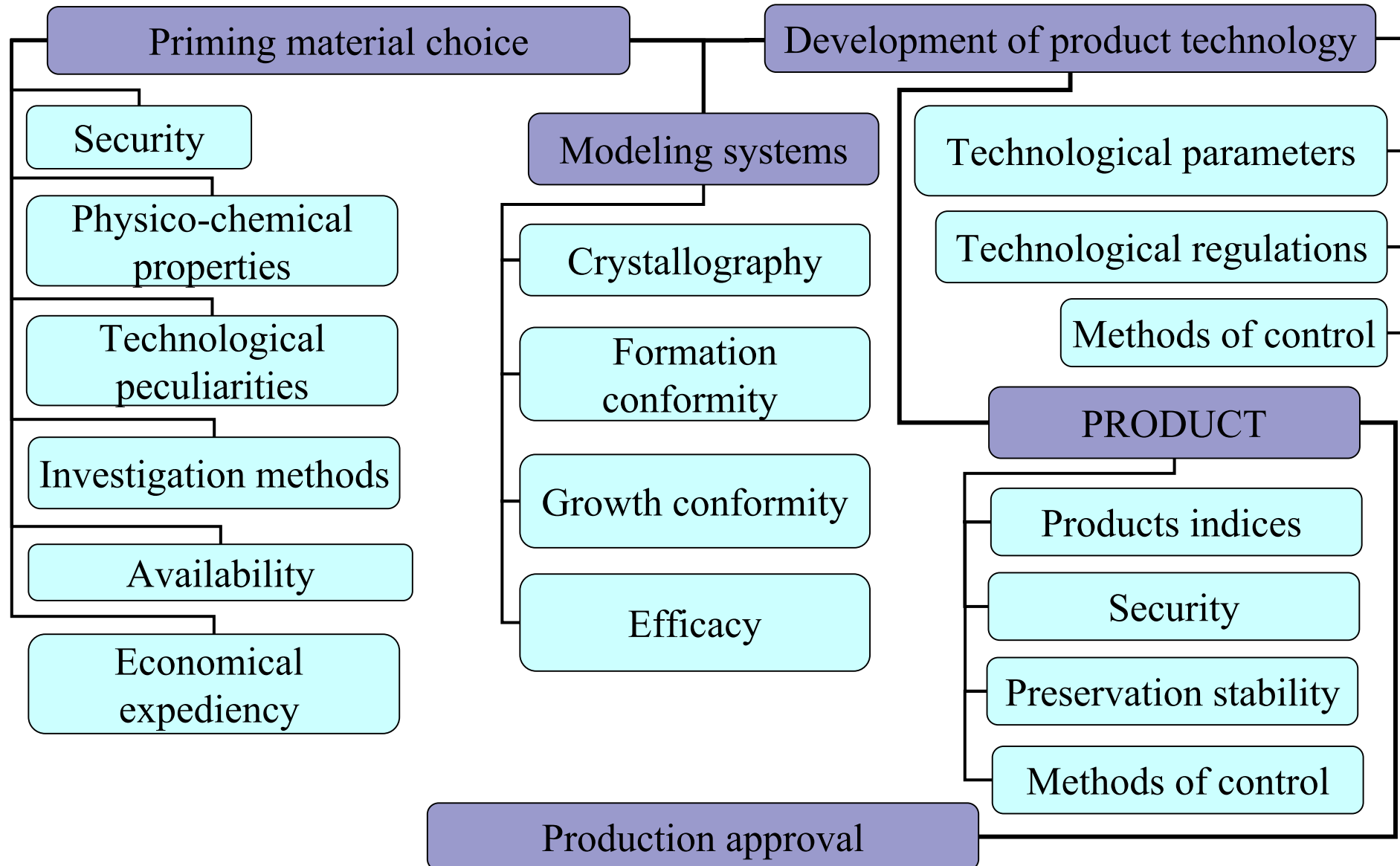
 $\text{SiO}_2 > \text{TiO}_2 > \text{CaCO}_3$ 

## Results of lactose crystallization depending on the operating model and priming material type

Priming material type (1-2 $\mu\text{m}$ )	<i>Variant of modification of germs adding time</i>							
	A				B			
	K, %	D, $\mu\text{m}$	U	M, in 1mm <sup>3</sup>	K, %	D, $\mu\text{m}$	U	M, in 1mm <sup>3</sup>
SiO <sub>2</sub>	0,082	6,41	0,76	770.000	0,051	6,65	0,82	500.000
TiO <sub>2</sub>	0,089	8,26	0,54	270.000	0,061	6,86	0,63	500.000
CaCO <sub>3</sub>	0,098	9,13	0,41	220.000	0,078	7,68	0,59	270.000
Priming material type (1-2 $\mu\text{m}$ )	<i>Variant of modification of germs adding time</i>							
	C				C'			
	K, %	D, $\mu\text{m}$	U	M, in 1mm <sup>3</sup>	K, %	D, $\mu\text{m}$	U	M, in 1mm <sup>3</sup>
SiO <sub>2</sub>	0,022	4,48	0,88	$\geq 770.000$	0,022	6.98	0.64	500.000
TiO <sub>2</sub>	0,029	5,67	0,85	$\geq 770.000$	-	-	-	-
CaCO <sub>3</sub>	0,031	7,09	0,82	500.000	-	-	-	-



# Farther investigations algorithm



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