State of work in the field of nuclear emulsion production
in the Slavich Company

(Oleg Orurk, Slavich Company)

Introduction

The Slavich Company is one of the oldest Russian enterprises in the field of production of photographic materials.

Since 1974, the Slavich Company organized production of high resolution photographic plates HRP for microelectronics based on high resolution nanoemulsion HR containing nanocrystals of 60 nm. Hhigh resolution photographic plates are intended for preparing the precision phototemplates in microelectronics, and also for the production master - holograms of living unit and high-quality reflection holograms. Since 1978 the manufacture of photographic plates for holography based emulsion PE-2 is carried out. To date no one foreign company has mastered methods of synthesis of PE-2 type emulsions for holography. For the last years improvement of technologies was carried out and the production of four types of photographic plates and films for holography was mastered. Now Russia is the only manufacturer and supplier of photographic plates and films for holography based on emulsion PE-2.

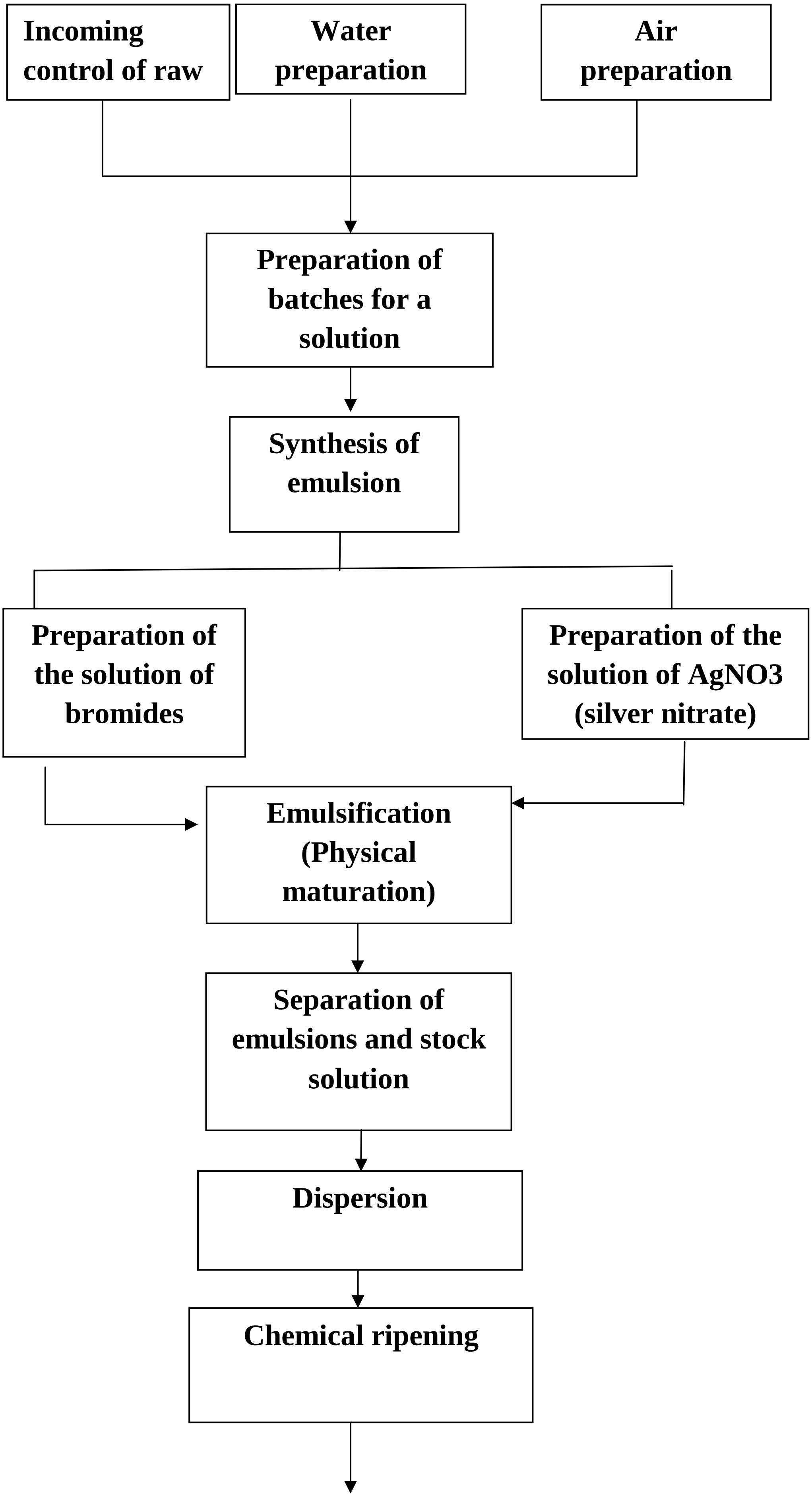
In 2008 in the Slavich Company on Micron workshop was initiated production of nuclear photoemulsions for scientific research. The main tasks set before the workshop was to restore the production of nuclear emulsions of P-2 and BD-2 types, as well as the creation of a new type of nuclear emulsion providing detection of particles with low energies with a precise definition of scattering. The development of the emulsion is supposed to use the known method of synthesis of holographic emulsion PE-2 with nanocrystals 8-15 nm.

At the moment, the Slavich Company remains the only Russian organization, which has preserved the line of production of nuclear emulsion. Here there was gained a great scientific and industrial experience in the nano-products development. The Slavich company has highly qualified engineers, researchers, engineers-technologists and workers with extensive experience in the development, improvement, and industrial production of photographic materials.

The photographic plate workshop Micron was put into operation on June 27 1975. It is a specific high-tech enterprise and has no analogues in domestic photochemistry. It was equipped under the contract with Japanese company "Wako Koyoki". Supervision was carried out by Japanese specialists with active participation of employees of the newly formed workshop. Joint efforts made it possible to create high performance photographic plates on which domestic microelectronics was originated and developed. In subsequent years production of 20 types of photographic plates for science, spectral analysis of ferrous and nonferrous metals, space research, astronomy, masks for color TVs were mastered on pouring conveyor. Micron workshop product in its photocharacteristics meets the analogs of foreign world famous companies. Some products are unique and have no analogues in the world. Photographic plates produced in Micron are widely used in the metallurgical industry of Russia and other countries. They bring unmatched beauty of holographic recording. They flew into space on board the spaceship «Buran» and in other devices. Micron astronomical plates with images of the night sky are a national treasure of Russia.

Actual state of works on nuclear photoemulsion

Fig. 1 shows a block diagram of technological process of nuclear emulsion production.


Incoming control of raw
Water preparation
Air preparation
Preparation of batches for a solution
Synthesis of emulsion
Preparation of the solution of bromides
Preparation of the solution of AgNO3 (silver nitrate)
Emulsification (Physical maturation)
Separation of emulsions and stock solution
Dispersion
Chemical ripening

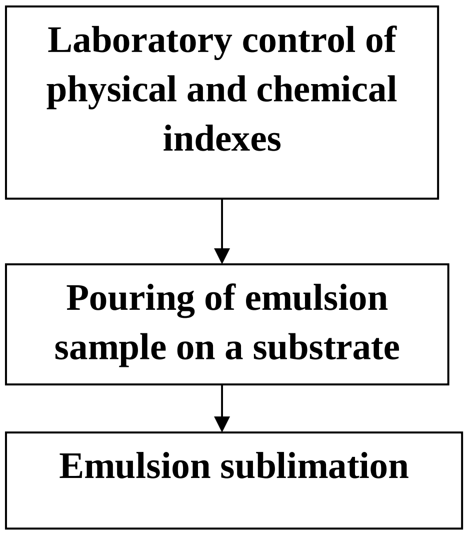


Fig. 1 Block diagram of technological process of nuclear emulsion production

All the experimental work is carried out in emulsion-found reactor with the capacity of 18 litres, presented on a photo 1.

Photo 1. Emulsion-found reactor with capacity of 18 liters.

From 2008 to September 2010 at the stage of separation of emulsion and mother solution the method of chemical deposition was used. Emulsions obtained by this concentration method had a significant dichroic fog and lack of sensitivity on the level of 15-20 seeds/100 microns. In September 2010 for R-2 emulsions there was initiated the technology of separation of emulsion and mother solution with the use of a special separator. It allowed to get rid of dichroic fog and to improve the sensitivity indicators. At the same time, in 2011 there started an intensive search for photographic gelatine test pieces from leading world manufacturers. Table 1 presents the list of producers of photographic gelatine, batch indexes and tested sample numbers.

Summary table of nuclear emulsion syntheses over the period from January 2011 to October 2013

Table 1

№	Manufacturer	Batch index	Number of syntheses
1	PB Gelatin (Belgium)	TS1647	3
2	PB Gelatin (Belgium)	L 9886	8
3	GELITA (Germany)	069146	11
4	GELITA (Germany)	149	2
5	Rousselot (France)	7400341S	2
6	Nitta Gelatin (Japan)	P-6420	2
7	Nitta Gelatin (Japan)	P-6421	1
8	Eastman Gelatine Corporation (USA)	37-668	1
9	Eastman Gelatine Corporation (USA)	2A	1
10	JSC Mojelit (Belarus)	brand P 17, batch 4871	1
	6 companies	10 indexes	32

Photomicrograph of AgBr microcrystals

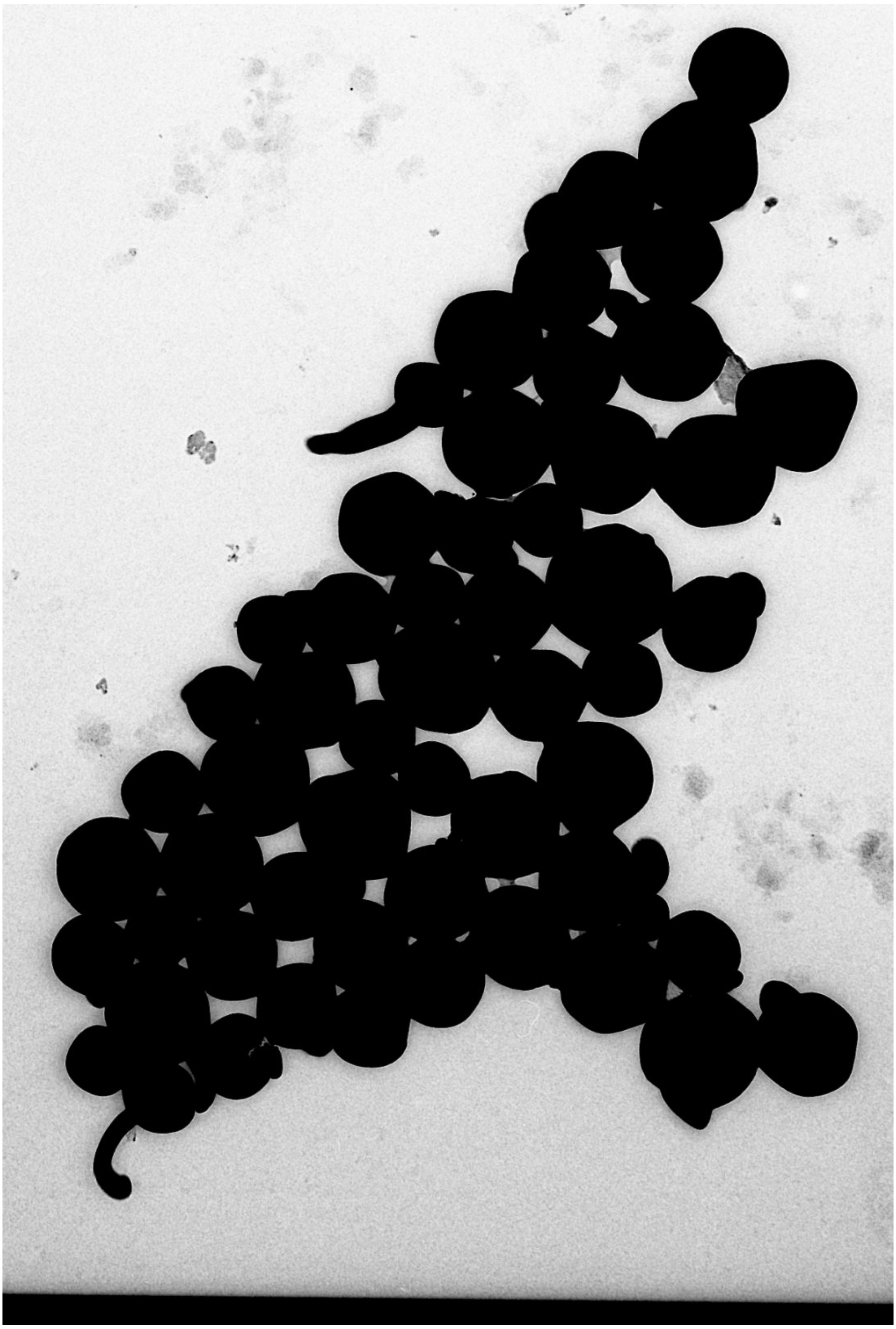


Fig.2 Slavich emulsion, batch 12C

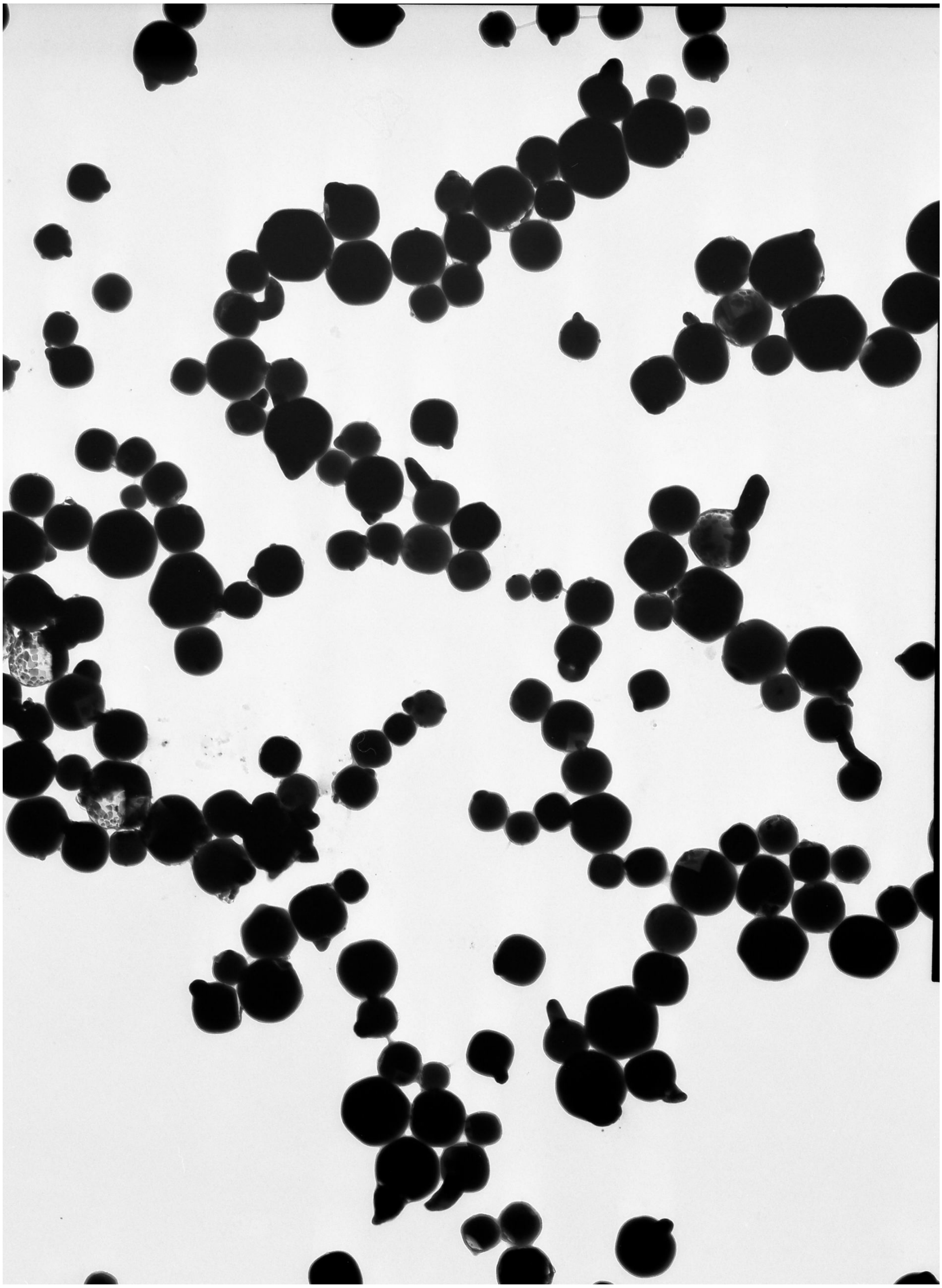


Fig.3 Slavich emulsion, batch 14C

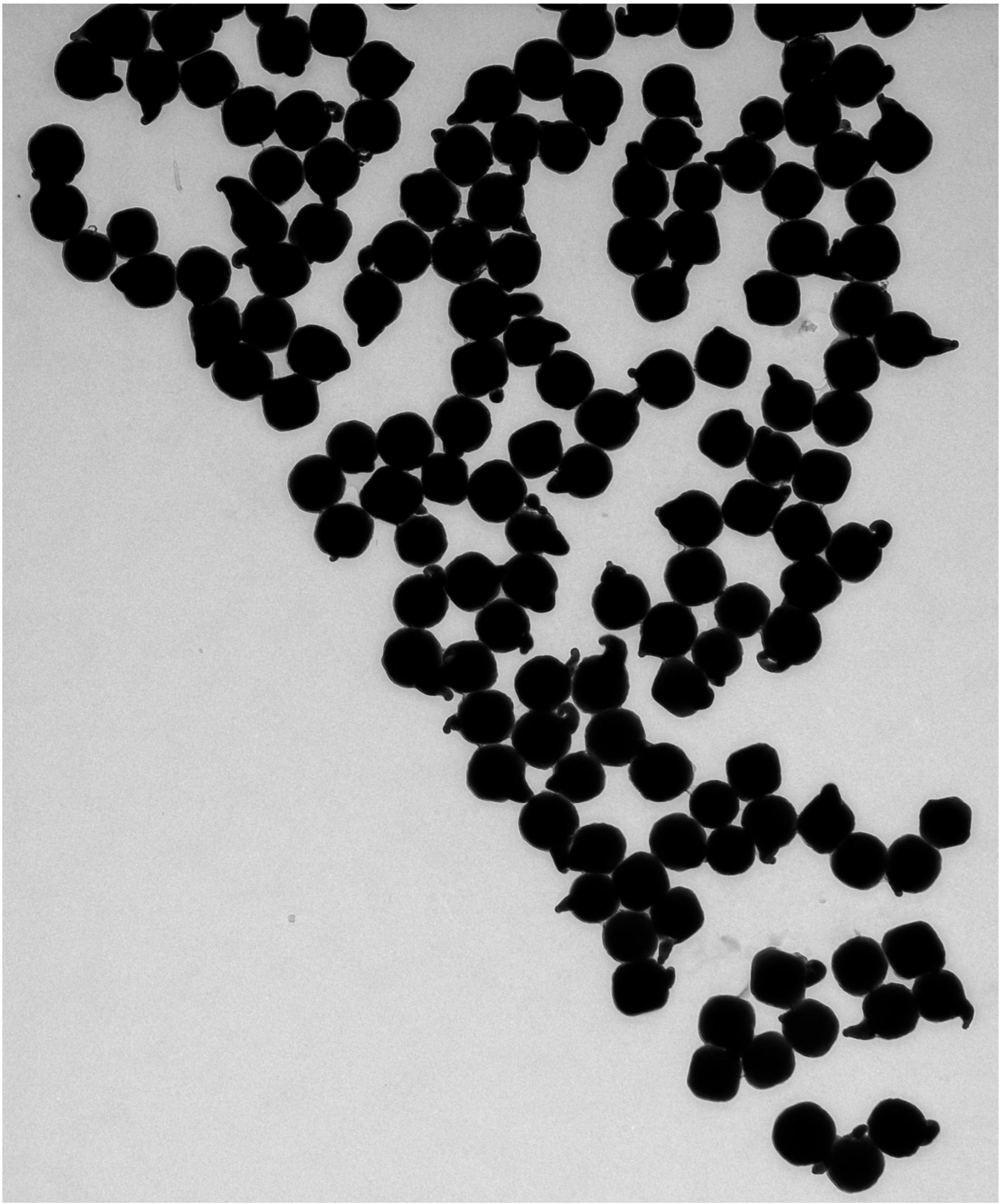


Fig. 4 Emulsion from Nagoya, Japan

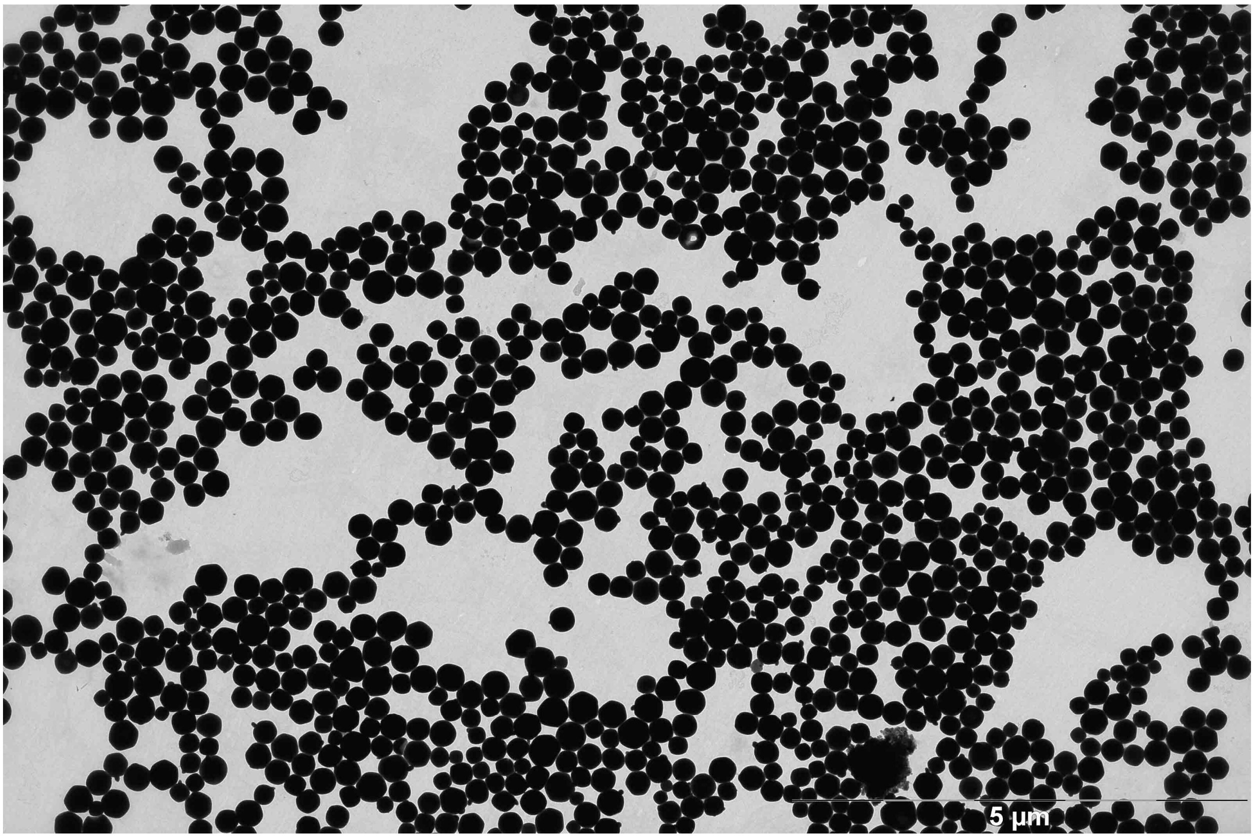


Fig. 5 Slavich emulsion, batch 38C

Microcrystal size distributions

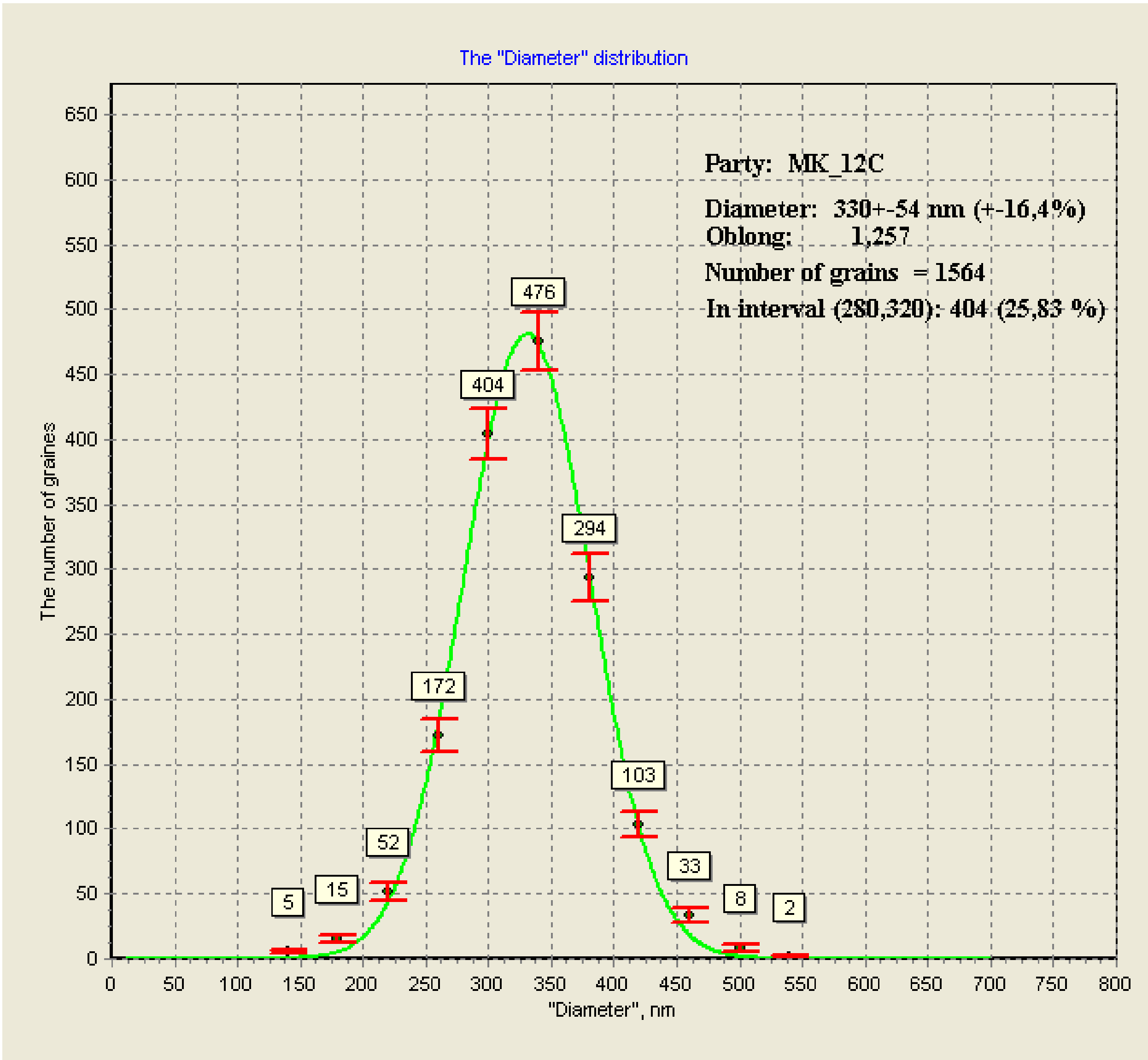


Fig. 6 Slavich emulsion, batch 12C

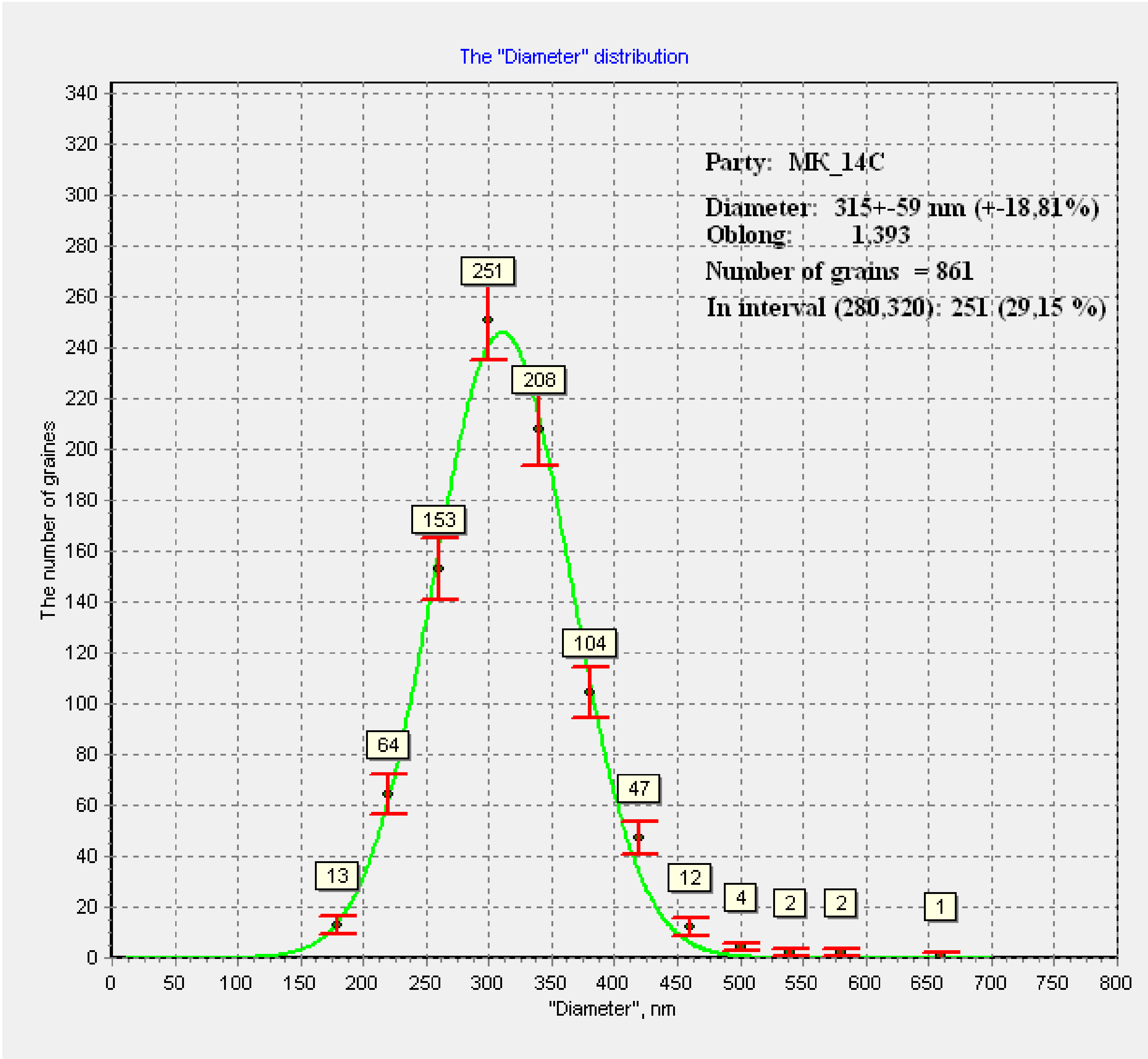


Fig.7 Slavich emulsion, batch 14C

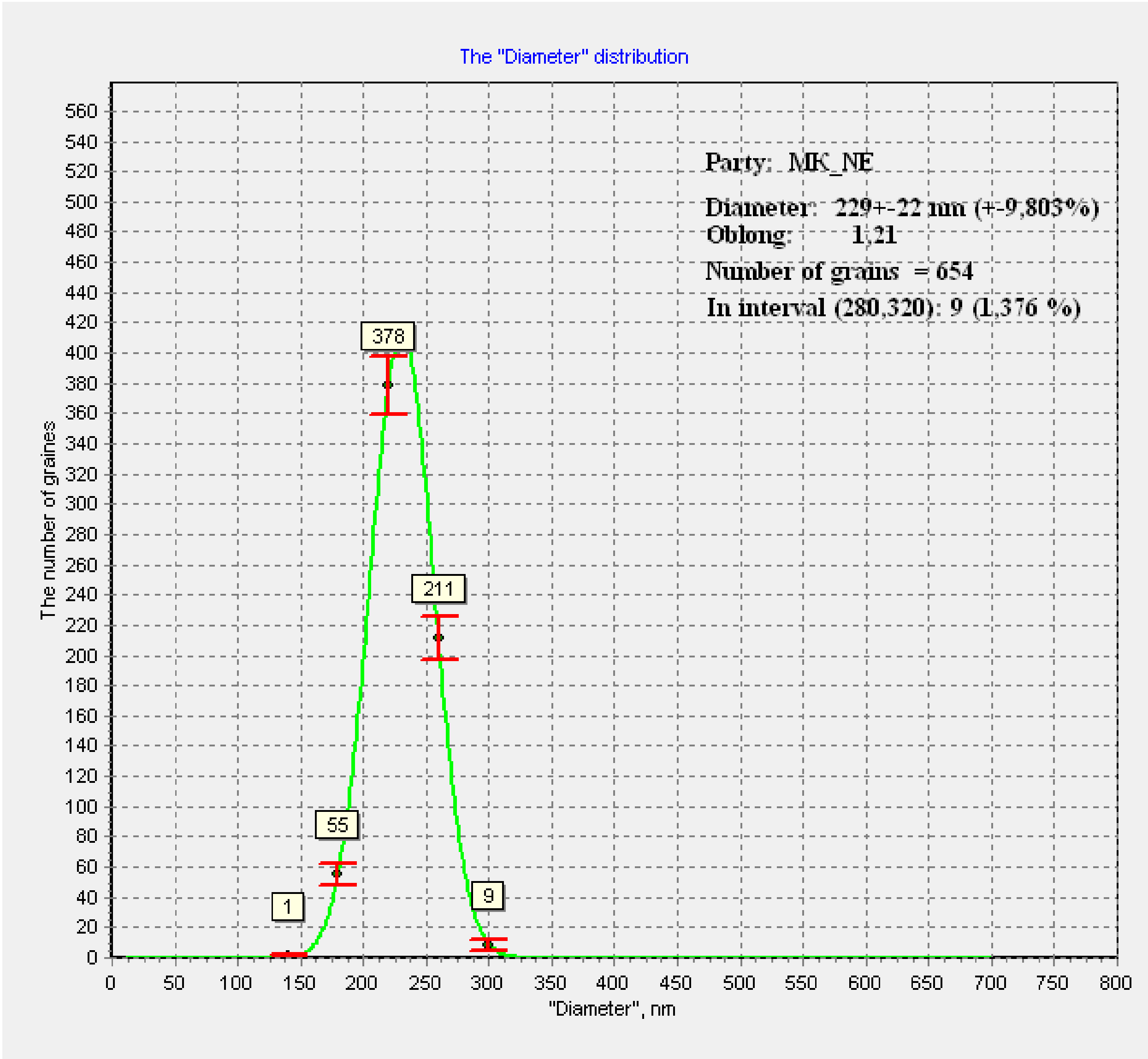


Fig. 8 Emulsion from Nagoya, Japan

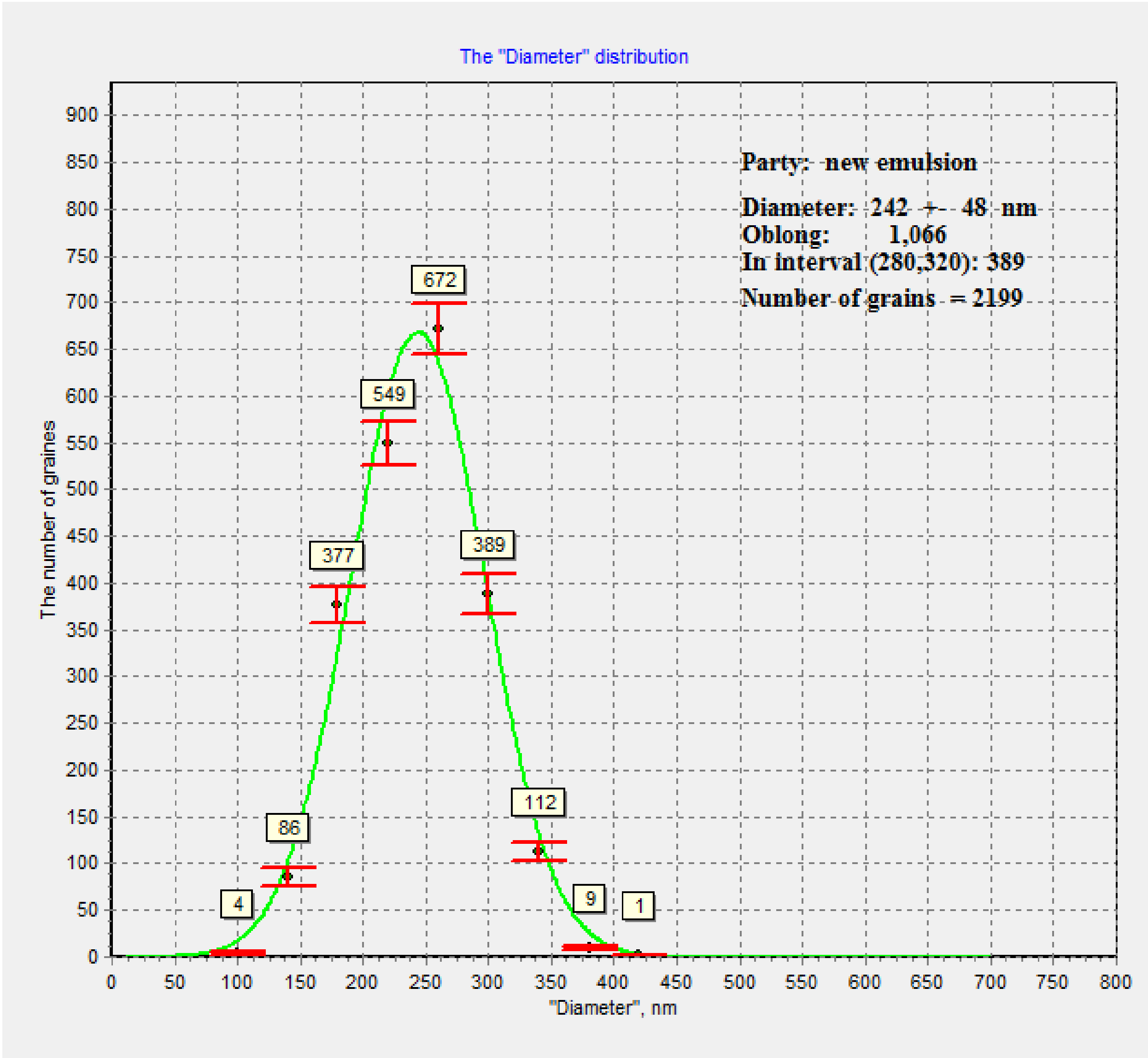


Fig. 9 Slavich emulsion, batch 38C

Comparison of the distribution

for various emulsion batch

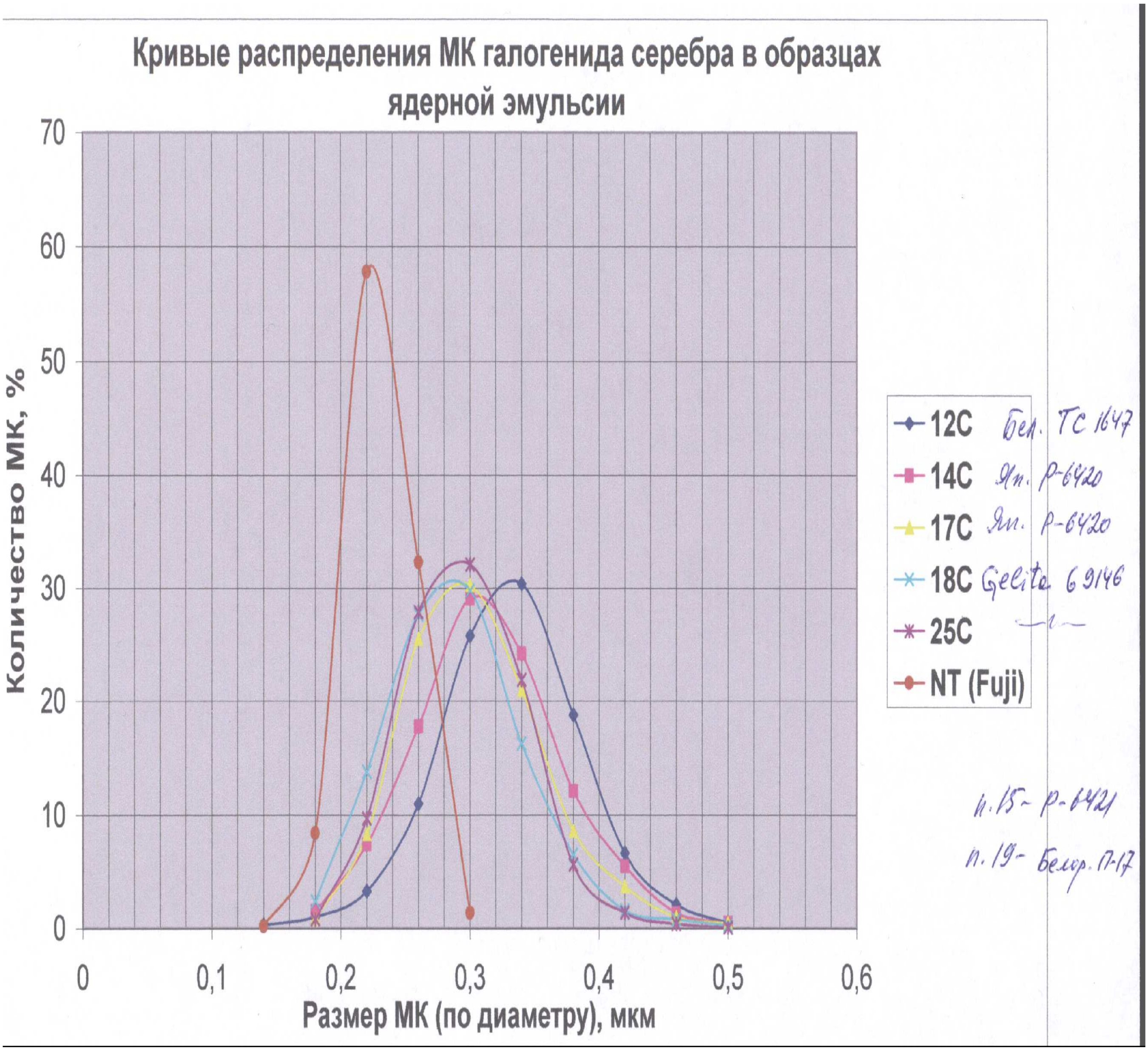


Fig.10

Comparative granulometric characteristics of AgHal microcrystals for some emulsion samples

Table 2

Batch	Number of observed microcrystals	Mean diameter, nm	ΔD, nm	Elongation (L/W)	Number of grains in the interval (280-320) nm	Share of grains in the interval (280-320) nm, %	Ionization (number of grains/100 microns)
NE Nagoya	654	229	22	1,21	9	1,38	No data
12C	1564	330	54	1,257	404	25,8	22
14C	861	315	59	1,393	251	29,15	22
17C	591	302	52	1,29	179	30,29	21
18C	1162	289	51	1,309	346	29,8	22
25C	1385	294	46	1,33	445	32,13	No data
28C	677	266	61	1,09	143	21,12	20
32C	705	297	52	1,064	193	27,38	22
38C	2199	242	48	1,066	389	17,69	No data

Comparative tests of Slavich emulsion (15C) and Japanese emulsion (Nagoya)

- **Nagoya film (OPERA developer, 20 min)**

point 1	point 2	point 3	point 4
30	35	35	32
39	36	36	33
37	40	32	42
33	36	30	35
Mean value of sensitivity is 35.06 grains per 100 mkm			

- **Slavich film, 15-th batch (OPERA developer, 10 min)**

point 1	point 2	point 3	point 4
25	25	24	29
28	21	25	27
29	23	27	25
30	28	26	23
Mean value of sensitivity is 25.94 grains per 100 mkm			

- **Slavich film, 15-th batch (OPERA developer, 20 min)**

point 1	point 2	point 3	point 4
27	28	30	25
29	26	28	27
23	28	27	29
25	23	25	28
Mean value of sensitivity is 26.75 grains per 100 mkm			

The data were obtained in the Laboratory for High Energy Physics, University of Bern (Switzerland).

Main conclusions

1. **Nuclear emulsions produced by the Slavich Company in their granulometric characteristics are nearing nuclear emulsions produced earlier in NIKFI - GOSNIIHIMFOTOPROEKT - PhoMos.**

2. **Some fluctuations of the AgBr microcrystal size distributions should not significantly affect the sensitivity of Slavich nuclear emulsions.**

3. **Silver content in the tested emulsion samples is brought up to the required minimum of 180 g/kg, what means that the number of AgBr microcrystals in the emulsion depth is sufficient.**

4. **The lack of sensitivity of nuclear emulsions produced by the Slavich Company may be a consequence of the fact that a fraction of AgBr microcrystals has no ability to record energy on the relativistic particle path. This may be conditioned by several reasons:**
   - the reduced microcrystal sensitivity insufficient for the particle energy registration;
   - ineffective development of this fraction of microcrystals, when the signs of the interaction with particles is weak.

Ways of nuclear emulsion production technology improvement

1. **Elaboration of optimal conditions for mixing of silver and bromides solutions.**
2. **Reduction of the average diameter and improving the AgBr microcrystal homogeneity by optimizing the type of mixer, mixing regimes and duration of synthesis (crystallization).**
3. **Optimization of conditions for the dispersion of sediment by the specification of the duration and mode dispersion of sediment in the water, selection of conditions of dispersed sediment fusion with gelatin, bringing the content of silver and gelatin in emulsions up to the required value.**
4. **Optimization of conditions for the silver halogenide solid-phase extraction and washing.**
5. **Optimization of the process of emulsion chemical maturation by optimizing the amount of gold and sodium thiosulfate.**
6. **Modernization of pouring division equipment for the purpose of mechanization of the emulsion pouring on glass, plastic, film.**
7. **Standardization of nuclear emulsion tests, selection of the best sources and modes of exposure, composition and modes of chemical-photographic processing.**

Thank you for your attention