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Possibility of using triphenylmethane  
as pelletized cold moderator of the IBR-2.

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## Introduction

Trends in the development of research in solid state physics are being directed to use in experiments, neutrons with a wavelength of more than 4 Å. Reducing energy fast neutron source it is carried out in a neutron moderator, which are "intermediaries" between the neutron source and the instruments of physical experiments. The required range of external neutron beams occurs both in thermal and cryogenic moderators, working at a temperature moderating material 20K-100K. The use of cryogenic neutron moderators gives increased output of "cold" neutrons, which in turn reduces the time required for a single experiment, and carry out those experiments that would not be possible without such a retarder because of their duration. Another, but no less important argument in favor of using cryogenic moderators is economic; since the use of cryogenic moderators produces comparable neutron intensity on weaker sources without building new ones, require huge expenses.

To slow the neutrons to thermal energies usually used ordinary water, the use of the majority of materials for cold moderators is difficult because of the problems of radiation resistance. In the existing moderators as moderating substance use ordinary water, liquid hydrogen, liquid hydrocarbons (methane, propane), a solid methane. Also possible to use water ice, frozen mixtures of methane and inert gas or unsaturated hydrocarbons, methane in zeolites, hydrates, methane, ammonia, aromatic hydrocarbons (such as trimethylbenzene (mesitylene)) and others. In the design of cryogenic neutron moderators, for sufficiently wide choice of materials it is necessary to give preference to any one material that would be most suitable for use. When you create a set of moderators for the neutron source is necessary to conduct preliminary research work, which consists in choosing the material, the thickness of the cold moderators themselves and premoderator, placement optimization, etc. The entire complex research allows more efficient use of the neutron source. These sources, in particular, is a research reactor IBR-2.

By the beginning of the XXI century. It was not yet sufficient information on the behavior of materials suitable for use as a working medium cold moderator (hereinafter CM) of neutrons in an intensive stream of fast neutron source of  $10^{12}$  n / cm<sup>2</sup>·s and above. In addition to liquid hydrogen, is not suffering from radiation damage and is used mainly on powerful accelerators (IBR-2 is not used for security reasons, and lower as compared to the yield of mesitylene cold neutrons).

Gaps in knowledge of radiation effects research was filled in a cryogenic irradiation facility IBR-2 [16]. In the first five years of this century at this facility were conducted extensive

studies of the effects of neutron irradiation on different materials for cold neutron moderators [17-20]. It was possible not only quantitatively clarify many of the effects, but also find a number of previously unknown, very fundamental to the practice of phenomena. It was found the phenomenon of spontaneous self-heating of the ice upon irradiation with fast neutrons at a temperature below 50 K, even at relatively low fluences of  $10^{16} \text{ n} \cdot \text{cm}^{-2}$ , and the detection of unusually rapid decline in the low-temperature thermal conductivity of the ice. When comparing the potential CM materials, such as methane and mesitylene (mixed, for example with m-xylene), it was found that methane is a poor resistance to radiation, as compared with mesitylene and high yield of hydrogen gas during the irradiation. A large amount of hydrogen causes the pressure on the chamber wall retarder repeatedly increases. To avoid failure, the wall thickness of the retarder chamber is increased, thereby reducing the intensity of cold neutrons from the surface of the chamber [25]. As a result of these studies, as the cold moderator of the IBR-2 was selected the appropriate material - aromatic hydrocarbon mesitylene in a mixture with m-xylene [26].

Currently, the LNP successfully operated CM neutron-based meta-xylene, and mesitylene. In addition, mesitylene conducted the study of neutron-physical properties of new materials, such as triphenylmethane (the TPM). It is worth noting that the TFM gives the same output of cold neutrons as mesitylene. At the same time, under the influence of irradiation in TFM is much less output radiolytic hydrogen [3] than the mesitylene. Another obvious advantage is the TPM that the material at room temperature is in the solid state (for example, a mixture of m-xylene and mesitylene - in liquid, gaseous, methane). Obtained from balls TPM not require special storage conditions (low temperature) and the transportation chamber retarder. For instance, the camera retarder they can be loaded at room temperature, and then cooled to the required cryogenic temperature.

According to preliminary theoretical estimates, the TPM may be longer than meta-xylene and mesitylene, work out as CM IBR-2. This is mainly because the yield of hydrogen from radiolytic TPM 10 times less than that of mesitylene [3].

Therefore, the purpose of work is to study the possibility of using triphenylmethane as a ball cold moderator of the IBR-2. In accordance with the purpose of the work, we were as follows:

1. Study of the radiation resistance of a mixture of meta-xylene, and mesitylene-TFM
2. Study the possibility of manufacturing the solid balls of TFM.

## ***Chapter 1 Overview of materials cold neutron moderators***

Consider materials, which currently are most commonly used as a neutron CM.

### **1.1 Liquid hydrogen**

On existing today in the world of the powerful neutron sources as CM most commonly used liquid hydrogen. Its main advantage - the absence of radiolysis and radiation effects. However, thermalization of neutrons in liquid hydrogen moderator is not complete. This is due to the lack of low-lying levels of excitement in the hydrogen molecule. In addition, hydrogen is explosive, which restricts its use. This is especially true for pulsed reactor, as he is ~ 40 times more sensitive to changes in the geometry than the reactors of stationary action. Therefore, hydrogen can not be used on the IBR-2 in connection with the problems of safety of operation of pulsed reactor.

### **1.2 Methane**

Methane is more effective to slow the neutrons, because its molecule has low lying rotational (rotary) levels. This, together with high density of hydrogen nuclei, makes methane neutron-physical characteristics of the best of all hitherto known substances. Yield cold neutrons solid methane at a temperature of 20 K was 2-3 times higher than that of hydrogen. Methane is also convenient because it can be used over a relatively wide range of temperatures. However, the main drawback of methane - a low radiation resistance. For example, the process of methane moderator at the IBR-2, 2-3 times a day had to change the temperature mode (for "forcing" the accumulated hydrogen), causing instability of the neutron spectrum. In addition, the service life of the methane moderator is small due to the accumulation in the cell pitches - solid radiolysis products.

Swelling of the matrix under the influence of hydrogen during heating radiolytic matrix and corresponding to that pressure on the chamber walls retarder depend not only on the concentration of the hydrogen accumulated but also on the rate of hydrogen release from the matrix.

### **1.3 Mesitylene**

As an active ingredient CM neutron sources for the average, power used mesitylene. Three properties of mesitylene are most attractive to use it as a moderator, especially in high radiation fields.

The first property mesitylene - high radiation resistance as compared with methane or ice. Comparative and absolute radiative properties are given in [3, 24, 27,28]. For example, the yield of hydrogen in mesitylene at 10 times lower than the methane and a mixture thereof with toluene or p-xylene - even 20 times. Accumulation radicals in mesitylene practically not, and thus there is no spontaneous bursts temperature, such as in methane or ice [18,21, 24,29].

The essence of the second property mesitylene - the presence in the molecule CH<sub>3</sub> three complexes that perform slightly hindered rotation around the axis of symmetry of the complex. The energy of the rotational oscillation is 7 meV in the solid crystalline mesitylene and 5 meV - in solid mixtures of mesitylene with other benzene derivatives, which dominates the structure of "proton glass" [30,31]. Such low-energy levels of molecules contribute rapid thermalization of neutrons. The available experimental data on both sections of inelastic scattering, and direct measurements of cold neutrons show that the material on the exit of cold neutrons is between the solid methane and liquid hydrogen and significantly better than ice.

The third positive property mesitylene - wide temperature range in which it is in condensed phases: a solid phase - below 228 K, and in the range from -45 to + 164 ° C - in the liquid phase. Loading and unloading of mesitylene convenient retarder cavity then at room temperature, and the temperature variation during operation to optimize the neutron spectrum in accordance with the requirements of the experiment. Another important factor is the fact that mesitylene is not explosive and less flammable than methane or hydrogen.

An important reason for preference mesitylene liquid hydrogen - is the complexity and high cost of establishing and operating a liquid hydrogen source as a result of the need to avoid hydrogen explosion. Policy highest reliability of the potentially dangerous radiation in terms of plants provides a triple shell liquid-hydrogen and helium circuit with vacuum cavity. This immediately leads to serious losses in the flow of cold neutrons. All of these factors are fundamental when choosing the material CM at the IBR-2.

#### 1.4 Some other materials

Besides the above materials in existing moderators as well as retarding substance used ordinary water, liquid hydrocarbons (propane [21]). Also possible to use ice, polyethylene, frozen mixtures of methane and other inert gas.

The LNP were first experimental studies of the processes of accumulation and recombination of radicals in solid methane, and other hydrogen-containing compounds when exposed to fast neutrons. Experiments were carried out on the installation URAM-2 was measured by the rate of

radiolytic hydrogen ice  $3.4 \times 10^{-8}$  mol / J (10% from the same quantity of methane) at 20-40 K and 10-20 MGy dose. [23]. There have been documented cases of spontaneous reactions of radical recombination (Figure 1 [18.24]). For the first time it has been shown that the phenomenon of spontaneous recombination reactions of radicals typical for many frozen compounds in which one of the radiolysis products are radicals. Of the substances studied reactions of radical recombination possible only in aromatic hydrocarbons.

Most susceptible to spontaneous reactions of radical recombination ice water at a temperature of less than about 40 K. The spontaneous heating of the ice to the 150-200K arose at a dose of 2-8 MG-(5-20 hours of fast neutron irradiated  $3 \cdot 10^{12}$  n/cm<sup>2</sup>/s). The introduction of some hydrogen acceptors little effect on the reaction conditions for the development of radical recombination.

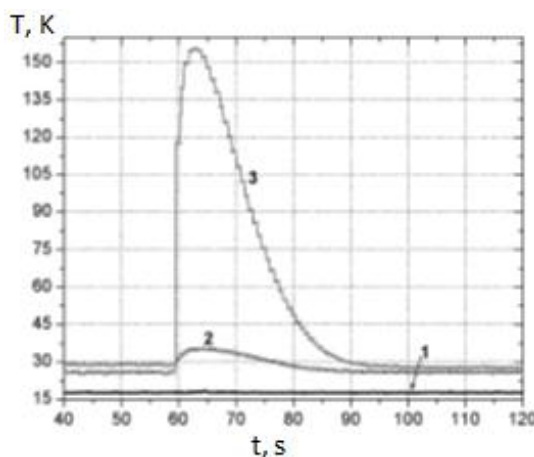


Fig. 1: The time course of the temperature in the spontaneous release of energy in the irradiated ice. 1- helium coolant temperature, temperature of the capsule wall 2 sample 3-ice temperature.

The research allowed determining the conditions of occurrence of both induced and spontaneous reactions of radical recombination, as well as patterns of accumulation of stored energy during irradiation. The most significant following conclusions:

- Energy accumulation occurs at a rate of 20-24 J / g / h in ice (5.4% -0.4% of the absorbed dose) and one hundred times more slowly - in not mesityl e
- Saturated latent energy density decreases with increasing temperature and is below the critical value (that allow rapid reaction of radical recombination) at a temperature of 45-50 K in ice.

- Contact the critical density of the radicals in which the possible induced "ignition" rapid response radical recombination with the ignition temperature is well described and methane ice, and Fig. 2 Rich P, this pattern does not depend on the size of the irradiated sample, at least for the larger sizes of 1-2mm.

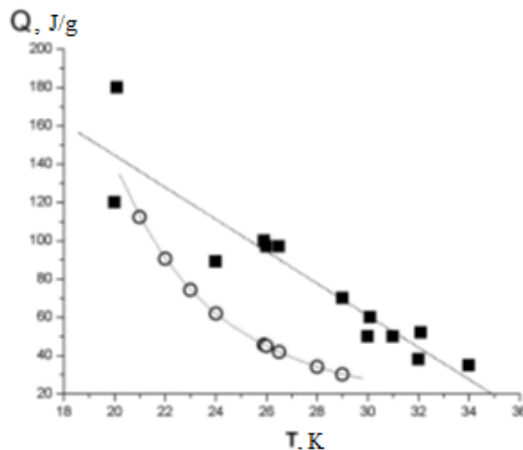


Fig. 2: Dependence of saturated density of stored energy in the irradiated solid methane  $Q$ , J / g, the irradiation temperature  $T$  R (circles) and the critical density of the stored energy from the ignition temperature (dark squares)

### 1.5 Triphenylmethane

TFM had never will be used as a cold moderator. Study its radiation resistance are fragmented. TFM (Figure 3) meets several key criteria for cold moderator:

- molecules consist of only hydrogen and carbon atoms.
- its absorption of neutrons can be considered low (5.4 barns / molecules for thermal neutrons)
- the hydrogen content in the range of a few percent of liquid hydrogen.

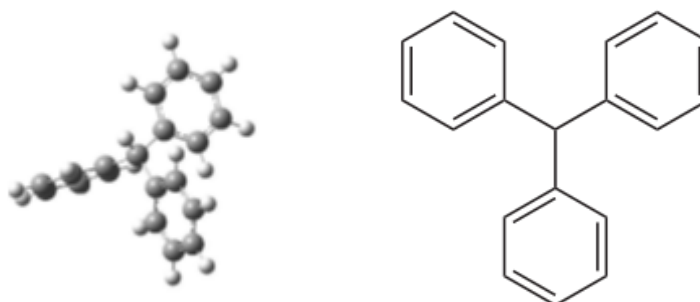


Fig. 3. Triphenylmethane molecule (left: ball and stick model, right: structure (hydrogen atoms omitted)).

These characteristics derive from its unique structure: as TPM in three aromatic phenyl groups surrounding a central carbon atom, in consequence, a molecule capable of forming relatively stable radicals or even ions. Radicals are usually quite unstable, and therefore creates a chemically aggressive environment in which the polymerization occurs. In aromatic



systems, the localized electron orbitals of several neighboring atoms overlap to form one huge orbital stretching over all atoms. This delocalized orbital is highly favored from an energetic point of view and lends outstanding radiation stability to molecules with aromatic carbon rings like benzene and mesitylene. Since energy C-H vibrations in aromatic molecules tend to be lower than their aliphatic analogues amount of high excitation in the material is expected to be significantly reduced. At the same time inhibited the axial rotation of the phenyl ring around the bond to the central atom, provides a plurality of low-energy excitation.

In general, the TPM may be a potential material for cold moderators, but it should be checked experimentally.

### 1.6 Problem statement

Of the above materials, it follows that the most suitable materials for CM IBR-2 reactor is mesitylene and TFM. During operation of the ball, CM based on m-xylene and mesitylene problems with increasing viscosity of the mixture at the end of the irradiation cycle, up to critical values. To solve this problem, you can either addition of various additives, or transfer to another material such as TFM. To understand the use of the TPM as a cold moderator of the IBR-2 is necessary to achieve the following objectives: 1) to study the radiation resistance of TFM compared to meta-xylene and mesitylene, 2) explore the possibility of making the solid balls of TFM.

### ***Chapter 2 Ball cold neutron moderator of the IBR-2.***

Consider the design and the concept of working the ball cold neutron moderator at the IBR-2. It is installed on one side of the hexagon of the reactor vessel for the neutron reflector. The complex (Fig. 4), besides the ball cold moderator include: water before the moderator, two water retarder and a comb-type postmoderator. Such a combination provides for a retarder of spectrometers on four neutron beams on that side of the reactor (7 th, 8 th, 10 th and 11 th beams) [1], the desired optimum range of cold and thermal neutrons. Luggage ball cold retarder made of an aluminum alloy AMG has internal dimensions to the front 150 x 200 mm and a thickness of 40 mm. Given the rate of filling the chamber balls 0.62, we obtain the effective thickness of the layer retarding substance 24-25 mm, which is optimal for the release of cold neutrons from the surface in the presence of water moderator a premoderator 50 mm [5].

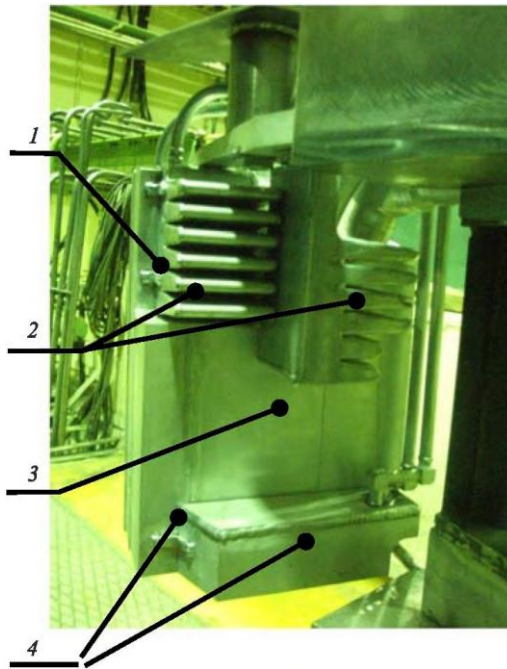
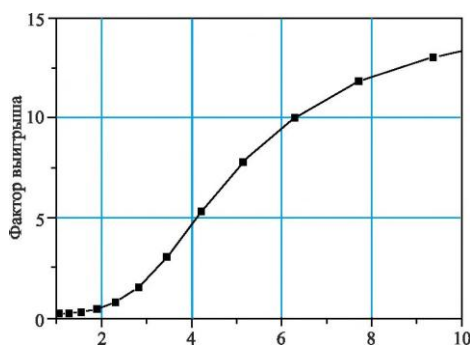


Fig. 4. Photo of the complex of neutron moderators at the IBR-2M in the direction of the beam number 7, 8, 10 and 11: 1 - water premoderator; 2 - water comb retardants; 3 - CM; 4 - water postmoderator

Thermal balls solid amorphous mixture of the meta-xylene and mesitylene slow absorption of neutrons and gamma rays -  $0.17 \text{ W / g}$ , the total power of heat in the moderator (including the chamber walls) reaches 160-170 watts. The retarder is cooled by passing downward through the filling balls cold helium from KSU-700 at a temperature of 25-27 K, while the average temperature in the chamber mesitylene moderator at a rate of cooling of the helium  $6 \text{ g / s}$  is 32-34 K. Estimated gain factor of the differential flux density of cold cold neutron moderator at 20 K compared to the traditional for the IBR-2 water-moderated comb type is shown in Fig. 5 [5].



Wavelength, A Fig. 5. Factor winning the cold neutron flux density with respect to water mesitylene moderator comb (calculation Monte Carlo)

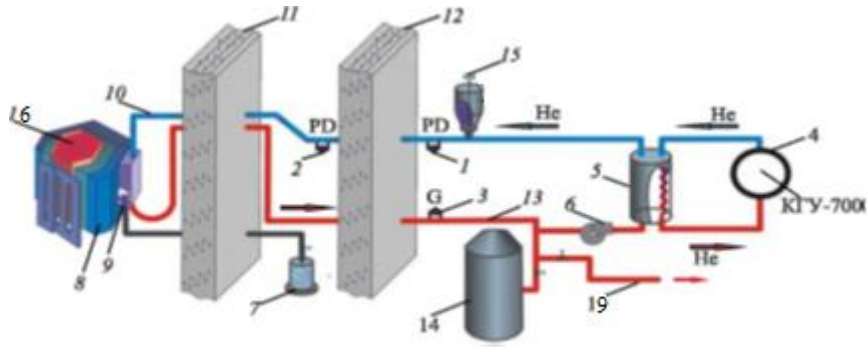


Fig. 6. Process flow diagram of the cryogenic moderator RS-202. 1,2 manometer; 3 Pitot tube; 4-KSU-700; 5- exchanger; 6-blower; 7-team capacity; 8-premoderator; 9- camera CM; 10,13- pipeline 11, 12 - of the reactor biological protection; 14 - Receiver helium gas; 15 dispenser; 16-IBR-2; 17 - Line reset radiolytic hydrogen atmosphere by heating balls.

Schematic flow diagram of the ball cold moderator of the IBR-2 is shown in Fig. 6. Loading of Beads retarder chamber (9) is carried by pipeline (10) a stream of cold helium at 80 K, the mass flow rate of 1.5 g / s (secondary cooling circuit). The circulation of the helium is carried out by means of the blower (6). Gas flow measurement is carried out using a Pitot tube (3). Cooling occurs when the helium as it passes through a heat exchanger which in turn cools the first circuit of helium cryogenic machine KSU-700 (4).

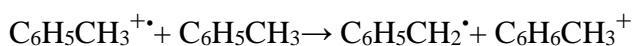
Solid beads mixture of mesitylene and m-xylene (3.5-3.9 mm diameter) are prepared in advance in a special unit and stored in liquid nitrogen. When loading they are poured into the dispenser (15), - an electromechanical device that provides quasi reset pipeline beads in small portions, an average of 2-4 in the second ball. Parameters helium carrier chosen so that the motion of balls on the complex path length of about 20 m with curves and steep rises, a large number of collisions with the walls of the tube retain their integrity and at the same time to avoid congestion. Loading chamber (approximately 27,000 balls) occurs in a time of about 4 hours. Tracking the movement of the ball is carried out by measuring the differential static pressure of gas from several points of the track pairs manometer PD (1, 2). In order to make sure that the camera is fully loaded moderator balls used a neutron imaging camera using the method of the camera obscura on a scale of 1: 2. This allows not only to see the arrangement of beads in the chamber, but also to remove the spatial variation of the energy spectrum of slow neutrons (using the method of time of flight), and hence, the temperature distribution retarding substance. After feed chamber temperature is lowered to 30-35 loop K and remains so until the end of the loop reactor. The mass flow rate of helium in this case reaches values of 5-6 g / sec. After the end of cycle the power of the reactor is reduced to 0, KSU-700 stops and the balls in the chamber begin to melt. Irradiated the expanded liquid is discharged from the chamber into a special container (7) and sent to storage as liquid radioactive waste.

### **Chapter 3 Using the mesitylene in a mixture of meta-xylene, and triphenylmethane as materials ball cold moderator of the IBR-2**

3.1 Results of operation for easy cold moderator of the IBR-2 with the loading chamber with a mixture of meta-xylene, and mesitylene.

We studied a sample of mesitylene - meta-xylene after irradiation in a real reactor experiment with a cold moderator. The sample was irradiated at 30 K to 217 MG-dose (8.6 days). Main way interest was the radiation resistance of the sample.

In general, it can be concluded that the radiolysis of m-xylene and mesitylene gap occurs predominantly C - H bonds (yield of hydrogen by approximately one order lower than that of methane), and the major end products are probably oligomers and polymers of unknown composition. At low temperature radiolysis, stabilizing radicals benzyl type (products of hydrogen abstraction from the methyl groups) and cyclohexadienyl type (adducts of hydrogen atoms to the benzene ring). Formation of the phenyl radicals of the type (product of separation of the hydrogen atoms of the benzene ring) securely fix failed. Primary radical cations of the compounds are likely to stabilize in the radiolysis even at 77 K. These particles can undergo recombination with electrons, and ion-molecule reactions with the formation of radical is benzene type. The occurrence of the last process by irradiation of a solid glassy toluene at 77 K was directly shown in [13]:



Information about the fate of excess electrons and the possibility of stabilizing the negative ions or the ion-radical completely absent. The excited states can decay with the formation of radicals such as benzyl, but the most likely is the dissipation of the excitation energy of radiative or nonradiative mechanism. The mechanism of the formation of oligomers and polymers is unclear nature of the dose dependence has not been studied, quantitative data on the radical yield virtually none. It is no information on the possible impact of additives aromatic compounds with lower ionization potentials and excitation mechanisms to radiolysis.

It is also worth noting a problem with increase in the viscosity of the mixture during the irradiation. Fig. 7a seen a sharp increase in the viscosity of the radiation dose to 10.2 days. This viscosity is critical to drain irradiated mixture from the chamber of the moderator. In this case, there is a high likelihood that a further increase in the viscosity of the irradiated mixture drains from the chamber, which will lead to serious consequences, and call into question the future work of the moderator in principle. Currently, a safe threshold increase in viscosity can be considered 8.5-9 days.

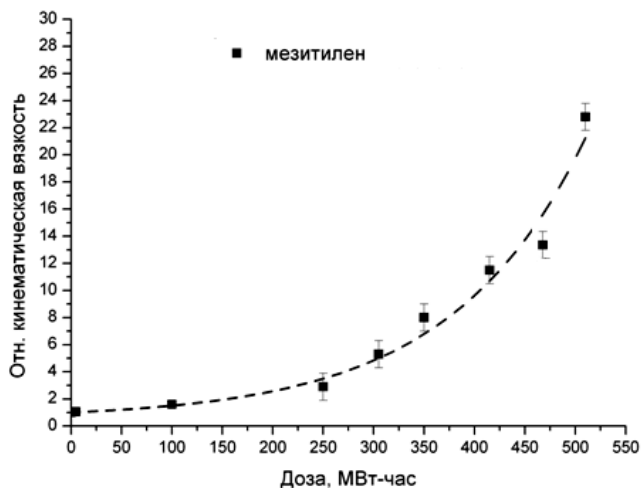


Figure 7. Change in the kinematic viscosity of the liquid mixture, depending on doses.

Fig. 7b mesitylene after 10 days of exposure.

On the other hand, according to the requirements at the time of reactor physics experiments at cryogenic retarder IBR-2 should be not less than 11 days. Therefore, in order to avoid further increase in viscosity were studied the possibility of introducing additives to enhance the work of the moderator to the desired setting (11 days). At the moment, we studied the addition of naphthalene - it gives a reduction in viscosity by 1.5 times, but because of the technological difficulties, and because of the difficulties that arise in the evacuation path of naphthalene after work retarder is not workable.

Thus, solutions using additives has not been found. Therefore, an alternative to the use of the TPM is very viable.

### 3.2 The published data on the inelastic scattering of TFM

In the Manual Lujan Jr. Neutron Scattering Center, experiments were conducted to study the neutron properties of TPM [2], this experiment has been modernized and is described in [33]. At Fig. 8 shows that the cross-section of triphenylmethane is almost independent of temperature above 30 meV. Below 30 meV however the total cross-section does show an increasing sensitivity to temperature. Between 0.3 and 30 meV the cross-section at 100 K reaches only about 60% of that at room temperature. This difference at lower energies can be attributed to the Debye-Waller factor. It can also be seen that the difference between the cross-section at 295 K and 100 K is significantly larger than the difference between 100 K and 10 K. These observations are consistent with those reported by LA Rodríguez Palomino for mesitylene and toluene [17]. In addition, it can be observed that all three cross-sections show several peaks in the range of 7-10 meV, which is an indication that the density of states for this material has some very strong modes in this energy range. To get an overview of the

compound's excitational modes, inelastic neutron scattering experiments were performed. Experimental spectrum shows The high intensity excitations in the range of roughly  $50\text{-}100\text{ cm}^{-1}$  ( $6.2\text{-}12.4\text{ meV}$ ), which corresponds to the energy range in question (Fig. 9)

Thus the scattering cross sections are consistent with meta-xylene and mesitylene [32], that is, the neutron-physical properties of the TPM is not worse mesitylene. Due to low energy excitations, which are so prominent in the spectrum supports our assumption that triphenylmethane potentially moderated to cold neutrons and can be used as CM IBR-2. However, this assumption is necessary should be verified experimentally.

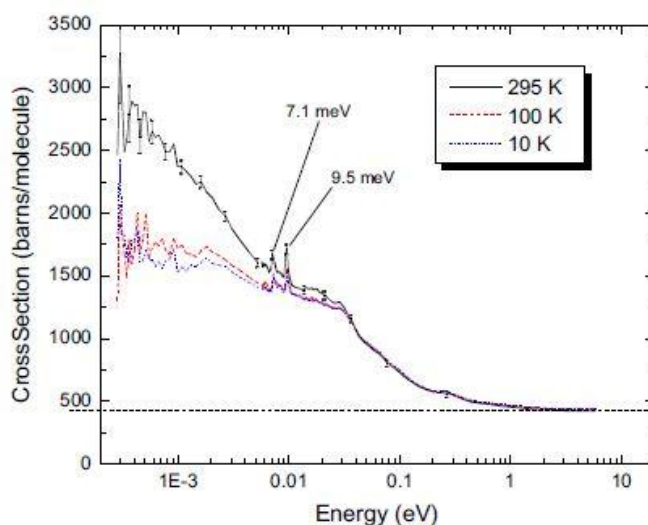


Fig. 8 Measured total neutron cross-section of triphenylmethane at various temperatures. The horizontal dashed line represents the calculated free gas model cross-section at 418 barns / molecule. Exemplary error bars are given for the 295 series K.

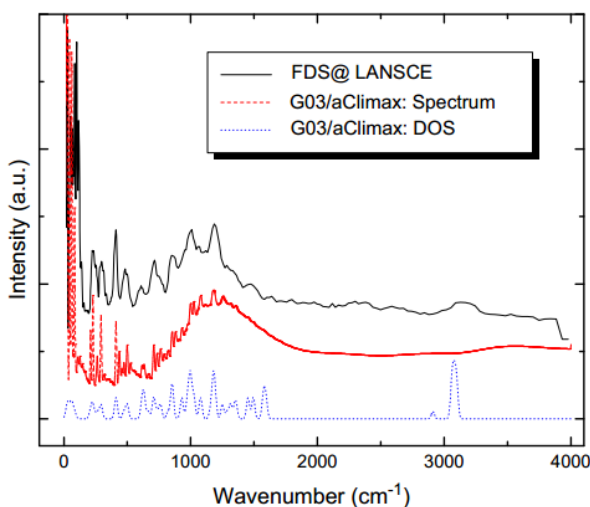


Fig. 9. Comparison between theoretical prediction (red) and experimental result (black) of the inelastic incoherent neutron spectrum. The blue graph shows the sum of the Density of States used in the calculations.

## Chapter 4 of the experiment proposal for a comparative study of the radiation resistance of a mixture of meta-xylene, and mesitylene with triphenylmethane at the IBR - 2.

As already noted, that the published data are very few, so in September 2015. It planned an experiment aimed at studying the radiation resistance of TFM. A study carried out on the radiation resistance of irradiation facility IBR-2 on the 3rd experimental channel.

### 4.1 Obluchatel'naya installation

Irradiation facility is a horizontal steel cylinder filled with water (the head portion) with a diameter of 800 mm and attached to the proximal end of the core to the I-beam transport (Figure 2). Samples for irradiation were placed in a metal container with the transverse dimensions not exceeding  $160 \text{ mm} \times 160 \text{ mm}$ , made of aluminum alloy AMG6. This material contains minor impurities, and gamma activity declined rapidly after exposure. The container is attached to the end of the I beam transport width of 100 mm, a height of 82 mm and a length of 2.7 m, also made of alloy AMG6. Transport girders connected to the thickest part of the irradiation facility with the Pomo soup collapsible flange connection. Irradiators moved along the track by means of a mechanism with the electric drive. Move the installation is limited by limit switches and emergency stops, located at both ends of the track, as well as the visible mechanical stop, which regulates the distance from the sample to the surface of the water moderator of the IBR-2 from the area of the 3rd channel in the experimental hall. Irradiation facility has a biological protection (Figure 10), protects personnel against ionizing radiation during operation.

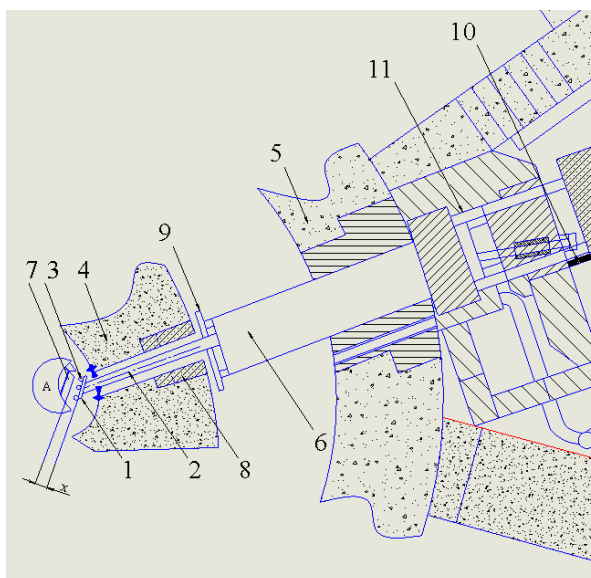


Figure 10. Driving irradiator 1 - metal container for fixing the samples; 2 - I-beam traffic; 3 - samples for irradiation; 4 - the first biological protection; 5 - the second biological protection; 6 - a massive part of the irradiation facility; 7 - water moderator; A - the active zone of the reactor IBR-2; X - distance from the moderator to the samples; 8 - Sliding biological protection; 9 - emergency stops; 10 - visible mechanical stop, 11 - track. All shaded elements in the diagram are the biological protection against radiation.

The irradiation time depends on the desired neutron fluence varies from a few hours to 11 days (normal duration of a session of the IBR-2). The temperature of the samples during irradiation does not exceed 50 ° C, which is partially provided by the system ventilation. If necessary to irradiate the samples at a cryogenic temperature. Control of the neutron flux is carried out by placing activation foils near the irradiated material and then measuring the induced activity. The massive steel cylinder of the irradiation facility placed curved pipes that allow lay cables for the power supply and output signals from the devices to be monitored during dialing a given dose.

A more detailed picture of the front of the irradiation facility is shown in Figure 11. The samples must not exceed the dimensions of the metal container (160mm × 160mm).

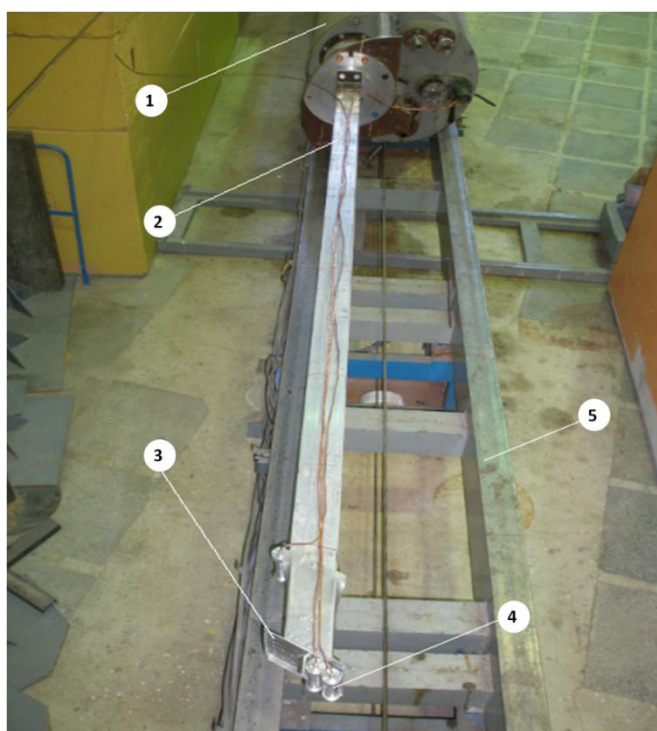


Fig. 11. irradiators channel №3 IBR-2 experimental hall, a view from the exterior of biological protection: 1 - a massive part of the irradiation facility, 2 - Transport girders, 3 - metal container for fixing the samples 4 - sample, 5 - railway way.

Neutron-physical characteristics of the irradiation facility of the IBR-2 are specified in [33]

#### 4.2 Statement of the experiment on the radiation resistance

Experiments on the radiation resistance differ according to different parameters. Arrangement of the experiment for radiation mixture of mesitylene and meta-xylene and TFM is shown in Fig. 12. The system consists of a capsule (1) for samples. Capsules are vacuum-tight lid and a cylinder, 70 mm height and 30 mm diameter. Capsules are made of alloy AMG6. Just experiment will use 3 capsules: one with mesitylene and meta-xylene, with the second and third



TFM polyethylene (reference sample). Each capsule is connected by means of copper pipes (2) having a diameter of 5 mm with a compensating volume (3). It is intended that the pressure at the pressure gauge during exposure does not exceed the maximum possible.

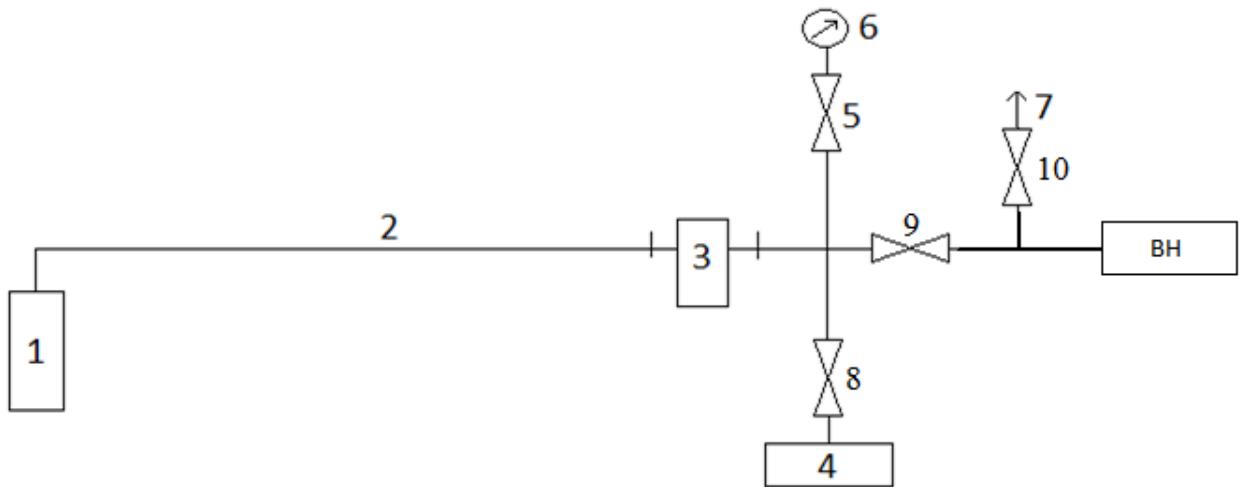


Fig. 12 Scheme of the experiment. 1 capsule with samples of TFM; 2 copper tube; 3 pressurizer; 4-cylinder with helium; 5,8,9,10-valves; 6-a pressure gauge, 7-pressure discharge to the atmosphere; VN- vacuum pump.

Compensating volume via the tee and the valve (5) is connected with a manometer (6) through a valve (8) and a balloon of helium through the discharge valve (10) to a vacuum pump (BH). Preparation of the experiment is as follows. In the first place with the help of a helium leak detector check if there are any leaks in the system. To do this, connect the leak detector to the system via the bleed valve (10). Vacuum pump forevacuum get in the system and using the leak detector vacuum method searches for leaks. Gates 5,8 - closed 9,10 - open. If a leak in the system is missing, then we reimplemented valves 9,10, turn off the vacuum pump and a leak detector. The test samples are placed into capsules. Opening the valves 5 and 8 so that the value of the gas pressure in the system is supplied from the cylinder does not exceed  $2/3$  of full scale pressure gauge. In such a state reserve system for 24 hours to test the integrity of the system under pressure. If integrity is all right open valve (10) and releasing the pressure. The position of valves before the experiment should be as follows: 5 - open 8-10 - closed. Move the installation to the core by a predetermined distance before the loop reactor. During the cycle, fix gauges. At the end of the experiment, we compare gauges, draw conclusions about the radiation resistance of the samples and the output of radiolytic hydrogen. Visual inspection of the samples. Thereafter, as a rule, carried out neutron activation analysis of samples of the satellite (NiCr) for determination of the neutron spectrum. Let us consider it in more detail.

#### 4.3 The method of neutron activation analysis for the determination of the neutron spectrum for test samples

In the course of the practice it has been mastered the method of neutron activation analysis. The purpose of spectral analysis of gamma-Ni-Cr sample is the determination of activity (Bq) or amount (atoms mol) of a radioactive isotope in the sample at a given time.

Ni-Cr target satellites are good material for a complete neutron fluence. Since the resulting isotope of  $^{58}\text{Co}$  have a relatively short half-life ( $T_{1/2} = 70,86$  days), as well as Ni-Cr is a cheap material. Sample weight was equal 0,0075g. The target was irradiated for 339 hours by fast neutrons ( $E > 1$  MeV), together with samples of polyethylene with meta-xylene and mesitylene. The analysis is performed on gamma spectrometer IGC 21 (Figure 13).



Fig. 13. Equipment.

For the analysis of the spectrum of a sample requires the following information:

- Spectrum (file format. Spk)
- The name of the desired isotope irradiation of the sample end time
- The time at which you want to determine the activity or the amount of isotope
- Height  $h$ , at where measured sample. If  $h = 0$ , then required also Sizes and the shape of the sample (circle, square)

Spectrum analysis begins with a decision on what the peak will be measured. To do this, the library data from the spectra (4) is required and its isotope peaks (energy values) with the highest yield of gamma rays in the decay. Then, in the spectrum are investigated these peaks, and among them is selected with the highest peak statistics undistorted other peaks (Fig. 14).

The selected peak is measured by its area (the number of events in the peak). To do this, the left and right of the peak in the spectrum put markers (double click left and right mouse button), and then clicking "Add ROI» program calculates peak area, its center, the background level at the peak, the error computation. For most calculations enough errors at the level of 1%. If approximation error exceeds 10%, the measurement is statistically unreliable.

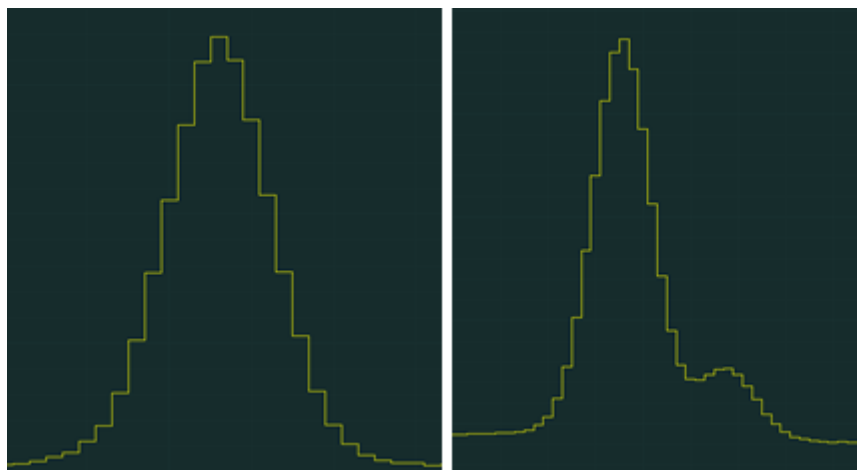


Fig. 14. Undistorted peak (left) and a peak distorted by the imposition of another peak (right).

When the values of all the above variables are known, the activity of the isotope in the sample at the measurement start time  $A_0$  is given by:

$$A_0 = \frac{N \cdot G \cdot D(T, T_{1/2})}{T_L \cdot \varepsilon(E, h) \cdot I_g}$$

In this formula  $N$  - peak area;  $E$  - energy peak;  $G$  - geometrical factor, other than units for the samples of extended, measured on the surface of the detector;  $T_L$  - live time;  $T$  - total time of measurement;  $T_{1/2}$  - the half-life of the isotope;  $I_g$  - yield gamma rays;  $\varepsilon(E, h)$  - the absolute efficiency of the spectrometer (measured and tabulated function);  $h$  - the height of the sample on a detector;  $D(T, T_{1/2})$  - correction due to the decay of the isotope in the measurement process:

$$D(T, T_{1/2}) = \frac{\ln 2}{(1 - 2^{-T/T_{1/2}})} \cdot \frac{T}{T_{1/2}}$$

The calculation is performed in the template implemented in the file Origin.

If the activity of the isotope (peak) is small, then the test is searched peak in the spectrum of the background. If the measurement of the background peak is detected the same energy, its activity should be subtracted from the activity of the desired isotope.

If activity is not necessary to calculate the beginning of measurement, and at another time, it is necessary to use the law of radioactive decay:

$$A = A_0 \cdot 2^{-\frac{t}{T_{1/2}}}$$

where  $t$  - time interval from the start of measurement till the time at which you want to find activity. If it is necessary to find activity in time before the start of measurement, the value of  $t$  is

negative. It should be remembered that the law of radioactive decay is only valid after the irradiation of the sample.

When the isotope activity  $A$  known amount of the isotope atoms,  $N$  can be found by the formula:

$$N = 1.4427 \cdot A \cdot T_{1/2}.$$

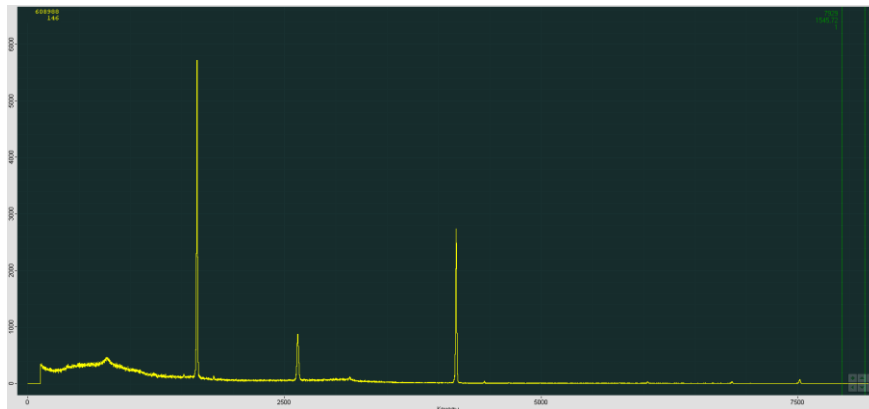


Figure 15. The spectrum of the irradiated sample Ni-Cr.

As a result, Ni-Cr measurement sample was prepared:

1. The activity at the beginning of the measurement - 72 818 Bq.
2. Activity at the end of irradiation - 102 146 Bq.
3. The flux density ( $> 1$  MeV)  $0.203172 \times 10^{12} \text{ n} / (\text{cm}^2 \cdot \text{s})$ .

### **Chapter 5 Learning opportunities hard balls of triphenylmethane**

Talking about the use of the TPM as a ball cold moderator of the IBR-2 is necessary to study the possibility of obtaining balls of TFM. Otherwise, talk about the ball-based moderator TFM has no meaning.

TFM at room temperature is in solid state in powder form. This can be considered an advantage by TFM compared with mesitylene as storing the resulting pellets after their manufacture to use is possible in every container at room temperature. Its melting point  $92\text{-}94^\circ \text{C}$  [15]. Therefore, you must first be heated TFM above the melting temperature and maintain this temperature throughout the time of receipt of balls. Several experiments were conducted to obtain solid spheres of the TPM with different fluids and different temperature of the environment.

The first experiments used in room temperature water as the coolant. Upon cooling, the drops in water at room temperature was observed the appearance of defects in the ball. It has been suggested that it is necessary to raise the temperature of the water and make a smoother transition of the ball from the air into the water. Require additional heating of the upper part of the vessel (ris.16b), the heating temperature reached  $76^{\circ}\text{C}$ , at the bottom of the water was room temperature. At ris.16b schematically shows an apparatus with water as the cooling liquid, which was conducted TPM obtaining pellets. In a glass flask was wearing a heater (2). The level of water (1) was roughly in the middle of the heater. Top located dropper surrounded heating elements (3). As a result of experiments it was observed the formation of a film on the surface of the TPM of water droplets which is subsequently flattened and with such geometry TPM can not be used as a moderator neurons. In consequence of the above, it can be concluded that the water used as the cooling medium can not.

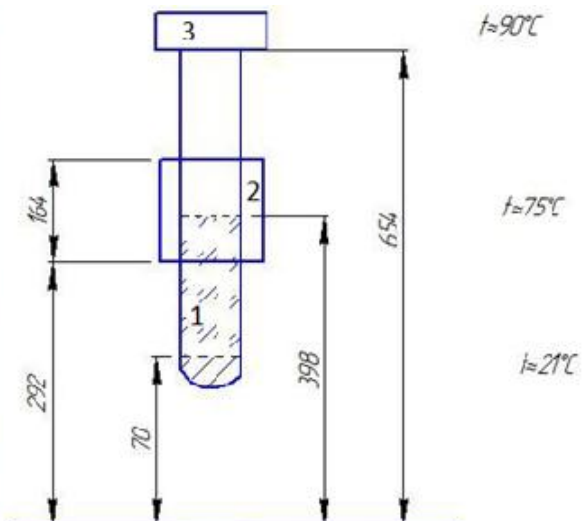
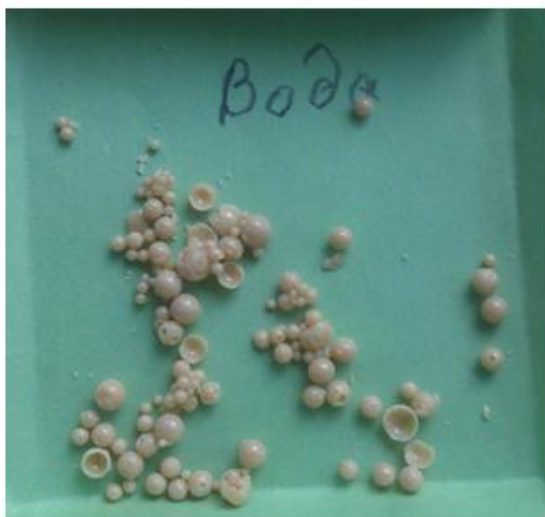


Fig. 16a. TFM balls chilled water Fig. 16b. The experimental setup. The coolant water.

It has been hypothesized that the surface tension of water too, so replace the water in the alcohol. When droplets hit the surface of the alcohol occurred spraying droplets at the entrance to the alcohol. What also does not meet the desired geometry (Fig. 17).



Fig. 17. TFM balls chilled alcohol.

As was the positive experience of using nitrogen in the preparation of mesitylene balls were carried out experiments with liquid nitrogen, and the clearance ranged from a drop of the dropper to the surface nitrogen 20mm and 150mm. Fig. 18a photo provided apparatus for producing TFM balls. The IV (3) was poured TFM heated to  $> 100^{\circ}\text{C}$ . The droplets fell into TPM Dewar vessel (2). The vessel was on a stand (1).

The beads obtained at a height of 150 mm had no regular shape (Fig. 18b). At a height of 20 mm balls get the right shape, though with a lot of marriage (Fig. 18b). This result should be considered satisfactory, but needs to be improved installation, to increase the number of balls and reduce the number of marriage.

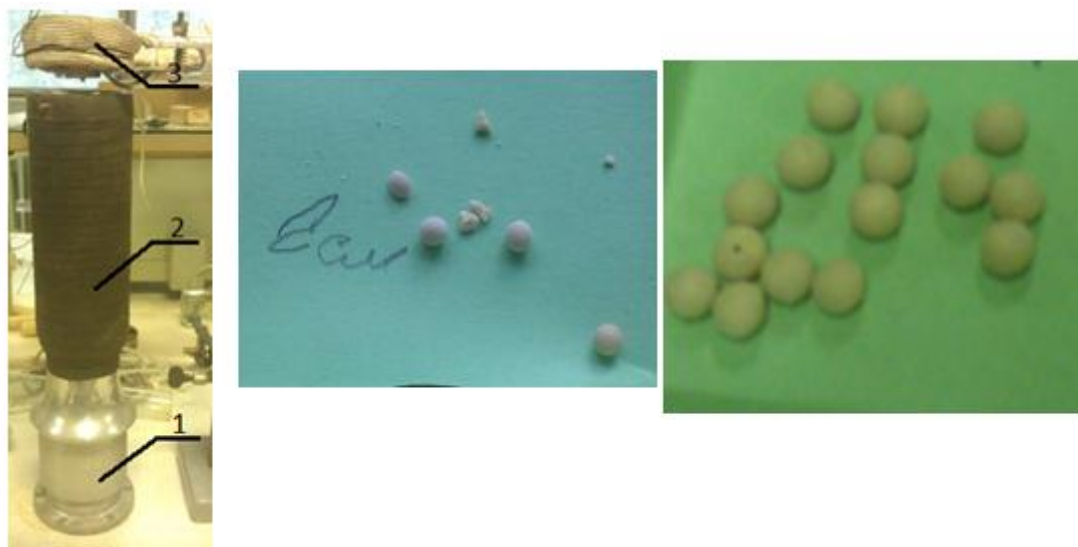


Fig. 18a. photo installation. Cooling liquid nitrogen; Fig. 18b and 18c TFM balls received in nitrogen.

The conclusions of the work

In the course of this work included a review of existing CM neutrons and analyzes the possibilities of their work on the IBR-2. In the result was made a choice in favor of TPM, as for

neutron-physical characteristics it is not worse mesitylene and theoretically it has better resistance to radiation.

To check the radiation resistance is necessary to carry out the experiment. For a quantitative description of the processes taking place in the TPM by fast neutrons, we need a method of neutron activation analysis for the determination of neutron fluence and spectrum of the samples.

To use the TPM as a ball cold moderator at the IBR-2, was examined the possibility of obtaining balls of TFM. On a specially created for this installation was shown the process of manufacturing the solid balls of TFM.

### Conclusion

Based on the above stated facts we can conclude that TPM may in the future become a working material for KZ202 IBR-2. In late September 2015. There will be an experiment to study the radiation resistance of TFM. It is also planned to construct a plant for mass production of beads of TFM. Conduct pre-loading beads to the chamber is scheduled for test bench cold moderator KZ201.

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