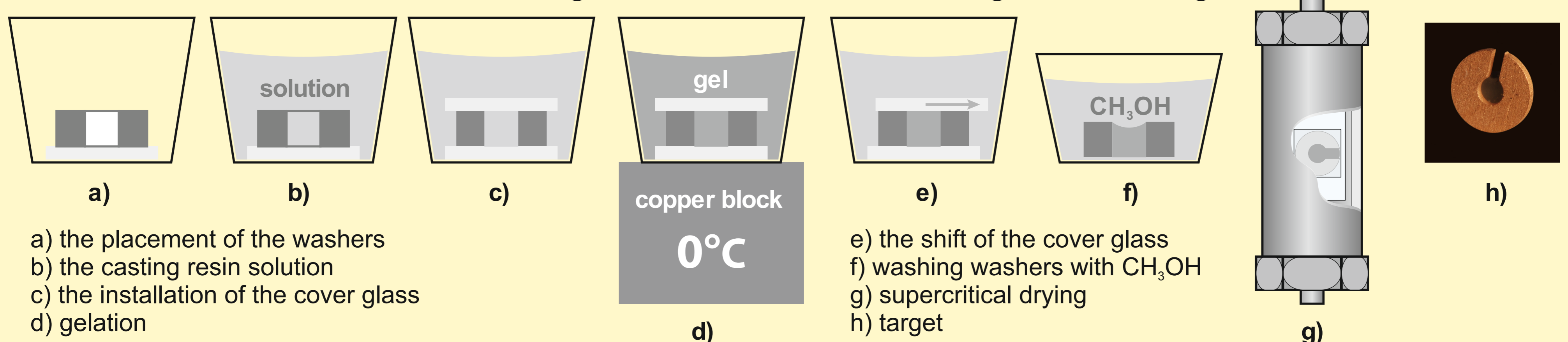




# Use of Freezing Techniques for Increasing the Output of Ultralow Density Laser Targets

V. G. Pimenov, E. E. Sheveleva, and A. M. Sakharov  
Zelinsky Institute of Organic Chemistry, Russian Academy of Sciences,  
Leninskii pr. 47, Moscow, 119991 Russia  
e-mail: sheveleva@mail.ioc.ac.ru

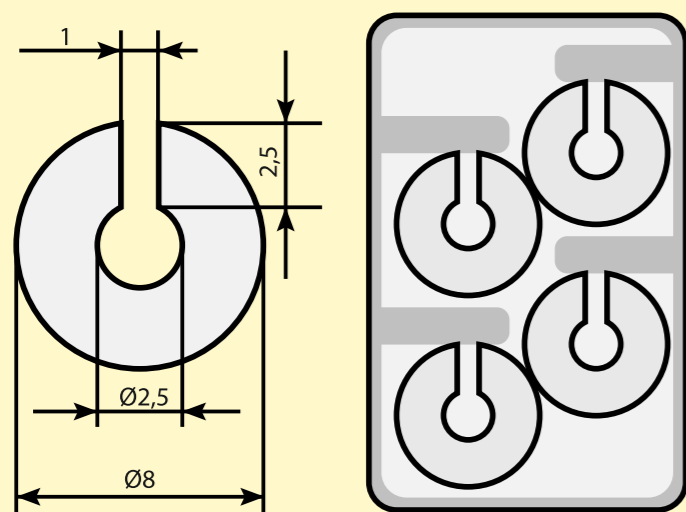
The traditional method of casting for the production of ultralow density microcellulose acetate laser targets with densities of  $2 \text{ mg/cm}^3$  and higher.



Aerogels obtained thereby are fully isotropic three-dimensional meshes with a 100% porosity. The separation between  $\sim 0.04\text{-}\mu\text{m}$ -diameter fibers is  $1\text{--}2 \mu\text{m}$ . The difference in the density between two adjacent foam regions  $30 \mu\text{m}$  in diameter does not exceed 1%. The pore diameter is  $2.8\text{--}20 \mu\text{m}$  at a density of  $1.4\text{--}8.8 \text{ mg/cm}^3$ , a specific surface area of  $125\text{--}280 \text{ m}^2/\text{g}$  according to the Brunauer–Emmett–Teller (BET) theory, and a porous layer thickness of  $\geq 60 \mu\text{m}$ . A simple method for obtaining a cast between glass slides consists in pouring a polymer solution over metal washers in the orifices of which a low-density microporous membrane will be fixed in position, applying glass slides, and initiating thermally induced gelation in the solution.

## Basic requirements to fill the low-concentrated solution.[1] The design features a snap.

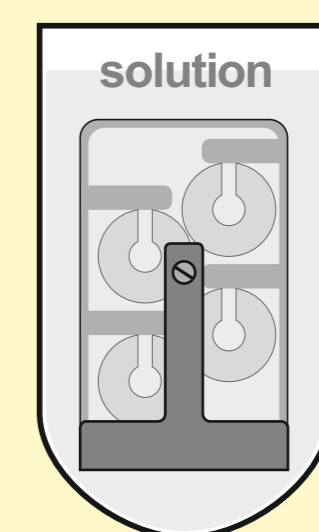
It was proposed drying the washers with a gel directly in a package between the glass slides and withdrawing the washers with a firm dry membrane as early as after drying.



Scheme 1  
Construction of the target



Scheme 2  
Assembly device for drying between the plates  
2(a) a pallet  
2(b) a glass slide  
2(c) the cover glass with recesses for drying between the plates  
2(d) a clamp  
2(e) stacking targets  
2(f) general view of the Assembly

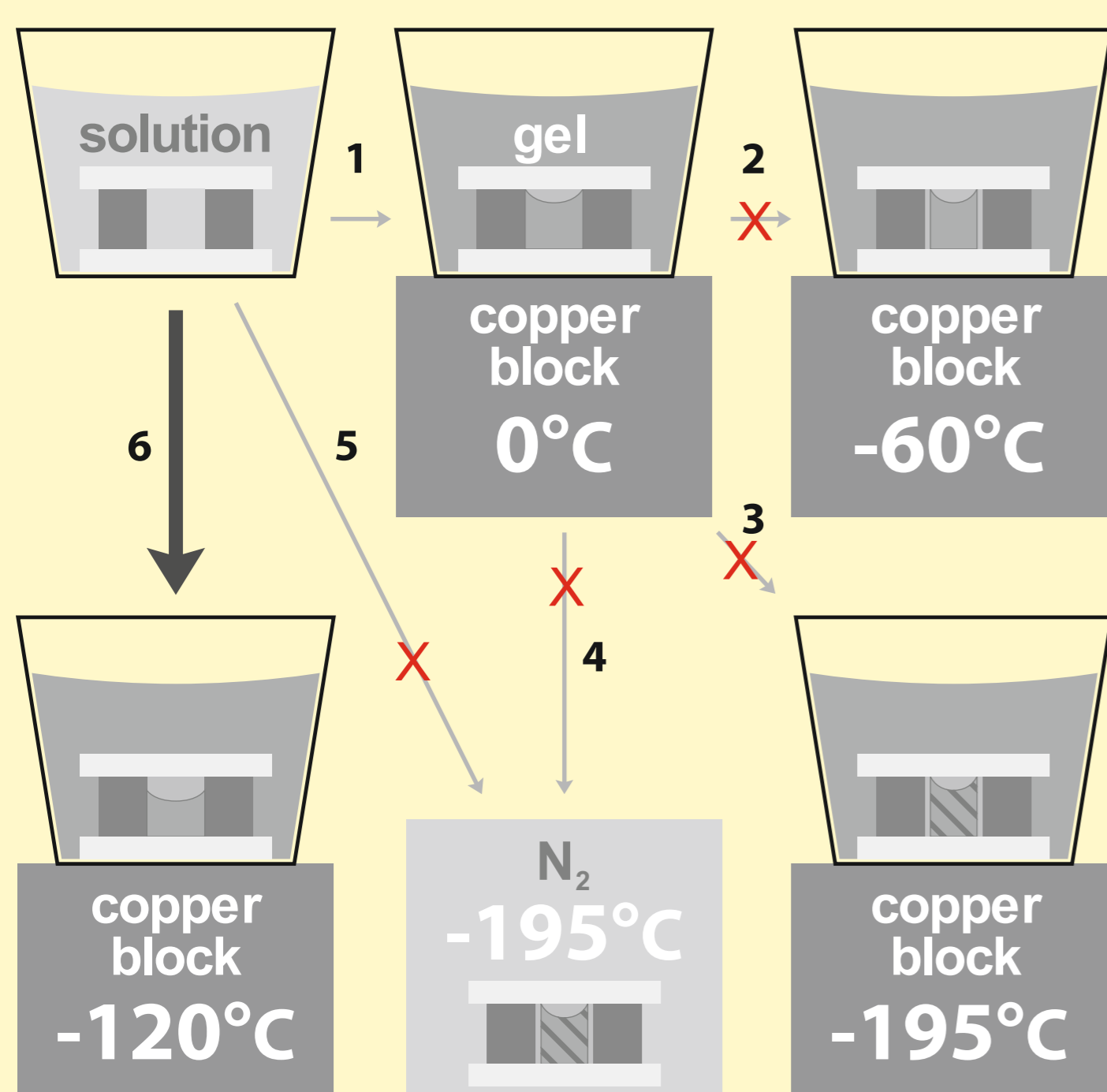


Scheme 3  
Casting with the use of a centrifuge

It was experimentally ascertained that the washers between the glasses can be dried if there is guaranteed access of carbonic acid liquid to the membrane and the diffusion depth of carbonic acid in the gel does not exceed  $4\text{--}5 \text{ mm}$ . For the access of carbonic acid to be guaranteed, a radial cut  $1 \text{ mm}$  wide was made in standard copper washers used to produce targets (outer diameter,  $8 \text{ mm}$ ; central hole diameter,  $2.5 \text{ mm}$ ; and washer thickness,  $60\text{--}500 \mu\text{m}$ ). Scheme 1. Before use, the washers passed standard treatment including chemical polishing, microprofiling, and chromate treatment. This was done to ensure maximum adhesion of a gel and an aerogel to a washer.

The gelling solution was prepared based on cellulose acetate and a mixture solvent (chloroform–methanol in equal volumes).

The package for casting was a set of four washers laid between glass slides, which, in turn, were fixed in place in a metal tray by means of a clamp. Scheme 2 The top slide had an intricate shape and recesses (see 2c). A centrifuge was used to pour a solution in a package (Scheme 3), since it was the only method for filling the washers with the solution in a narrow gap between the glass slides and avoiding evaporation of the solvent.



A dry aerogel with a density of  $2 \text{ mg/cm}^3$  has so low strength that, when adhered to any surface, it cannot be removed without damage. From this, it follows that, during molding, as a solution turns into a gel and, afterward, into an aerogel, the membrane being formed must stop touching the glass slides between which it was cast. In this connection, our attention was attracted to the phenomenon of polymer film self-peeling from the substrate.

It was ascertained that both syneresis and thermocyclic processing are generally useless or even detrimental. The point is that even a slight supercooling of washers with the gel (by a few degrees) with respect to the temperature at which gelation occurred leads to a break of the contact between the gel and the washer with the formation of a visible gap between them along the entire coupling periphery. Therefore, self-peeling, which leads to an increase in the target output, has not been attained either with the use of syneresis or heat treatment.

This problem was resolved by generating thermal stresses simultaneously with the formation of a gel and a membrane using considerably lower temperatures. Comparative freezing of solutions and gels revealed that, in the case of a solution, membrane-to-washer contact was not broken, and no gap between the washer and the membrane was observed. Moreover, production of targets using the initial solution and omitting the stage of preliminary gelation has led to a significant increase in the output, which grows in value with a decrease in the freezing temperature.

**After optimization, rapid freezing of a solution on a brick cooled to  $-120^\circ\text{C}$  was assumed to be the key point of the technique. Further processing has allowed the target density to be lowered to  $1.3 \text{ mg/cm}^3$  (thickness,  $150 \mu\text{m}$ ; output, 75%) and  $1.7 \text{ mg/cm}^3$  (thickness,  $100 \mu\text{m}$ ; output, 50%). Later on, the use of a block with an increasingly lower temperature has allowed us to attain a record-breaking density of  $1.0 \text{ mg/cm}^3$  at a washer thickness of  $150 \mu\text{m}$ .**

Contrary to this technique, rapid freezing changes the character of gelation from volumetric to layer-by-layer. A mold may simultaneously contain layers that have not got cold (at the center), layers in which the breakdown time has elapsed and conditions for gelation initiation have developed (intermediate layers), and layers composed of a just formed gel (at the bottom and the mold walls). Since a solution that has not changed to the gel state retains its mobility, it fills interstices and cracks produced upon system contraction, thus “curing” all defects that can be generated in a target. Incidentally, the target integrity is not affected, and a low freezing temperature guarantees formation of a gap between the upper glass and the gel.

**It is the self-supporting targets that have been manufactured for the first time with a density of  $1 \text{ mg/cm}^3$  and a window diameter of  $2.5 \text{ mm}$ , which is particularly important for a number of laser experiments. [1]**