

MS42-P136 - LATE | IN SITU CRYSTALLIZATION OF THE VISCOUS ORGANOSILICON LIQUIDS

Volodin, Alexander (INEOS, Moscow, RUS); Korlyukov, Alexander (INEOS, Moscow, RUS); Smo'yakov, Alexander (INEOS, Moscow, RUS)

This work presents the results of a study on the structure of various organosilanes and siloxanes. These substances are widely used in synthetic organometallic chemistry and in industry. Unfortunately, at room temperature, they are viscous liquids, that makes impossible to establish their structure using conventional X-ray diffraction studies. Such viscous compounds do not crystallize with slow cooling. For their crystallization, only shock freezing is appropriate. To establish the crystal structure of such compounds, we assembled the device of the zone melting of a crystal in a capillary with nichrome wire as heater. The features of this device are its simplicity and low cost, if we draw an analogy with commercially available OHCD (Optical Heating and Crystallization Device). In addition, being the authors of the device, we can freely complement and modify it.

Using this device, we were able to obtain previously unknown structures of more than a dozen of organosilicon compounds, which are liquids at room temperature. For some of the compounds studied, several crystalline phases were established. For example, the structure of octamethylcyclotetrasiloxane (D4) crystals at 12 different temperatures. A subsequent study of these structures by quantum chemistry methods made it possible to establish the most probable path of the solid-phase phase transition in the D4 crystal.