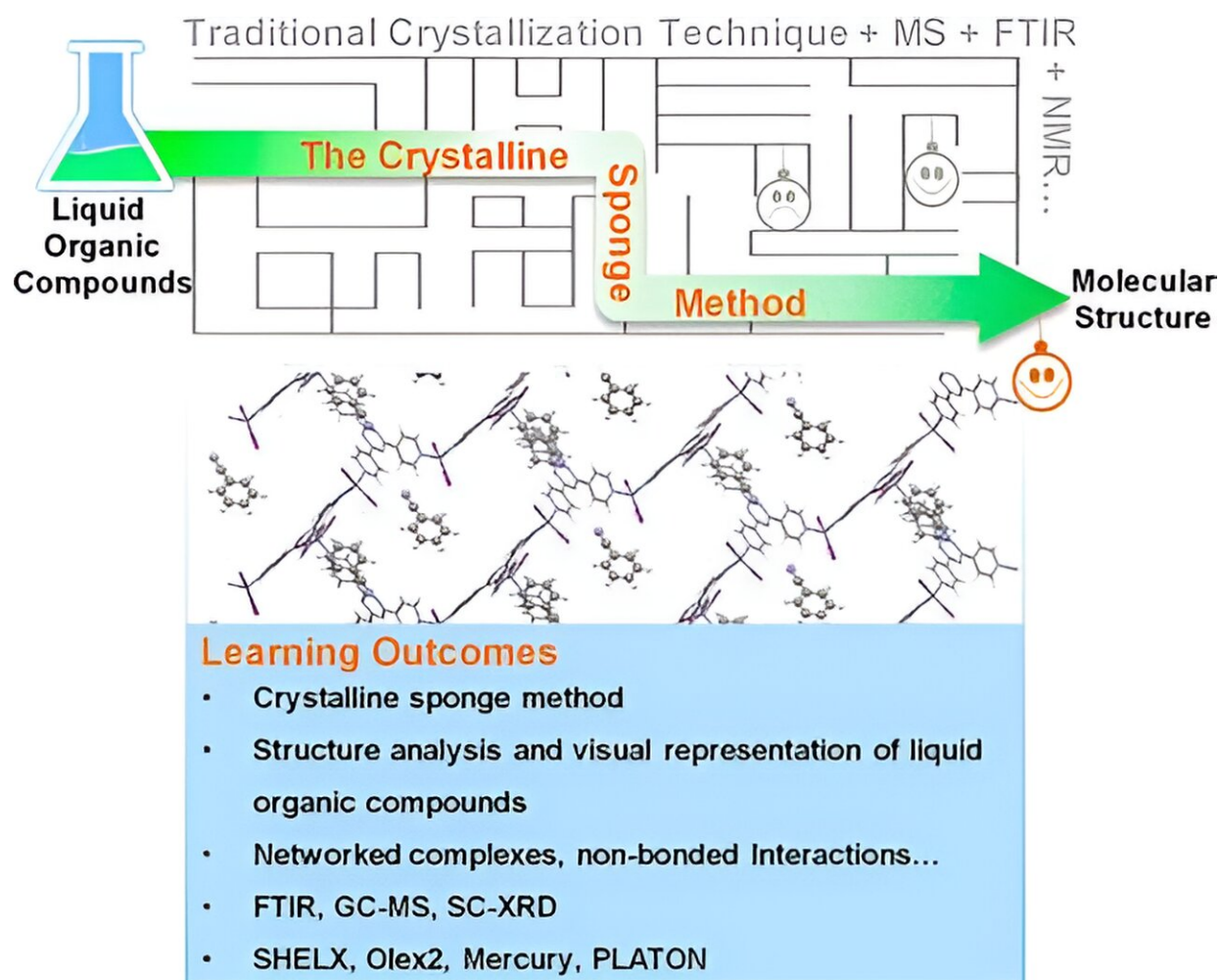


# New crystalline sponge method proposed for undergraduate courses

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The crystalline sponge method allows for direct and precise molecular structure determination of liquid and gaseous targets and thus has been recognized as a revolutionary breakthrough in crystallography. To expose undergraduates to this cutting-edge technique, we have developed a comprehensive laboratory

experiment with reaction conditions and characterizations systematically tailored for students to perform in a mild and accessible way. In this experiment, students investigate the preparation of networked complexes

$\{[(\text{ZnI}_2)_3(\text{TPT})_2] \cdot x(\text{solvate})\}_n$  as crystalline sponges with benzonitrile, methyl salicylate, and (trifluoromethoxy)benzene as solvent, respectively. Crystalline sponges obtained in benzonitrile were exposed to solvent exchange in cyclohexane to afford  $\{[(\text{ZnI}_2)_3(\text{TPT})_2] \cdot x(\text{cyclohexane})\}_n$ , and the progress was monitored by IR and GC-MS. All four crystals were evaluated under a microscope and subjected to single crystal X-ray diffraction (SC-XRD) analysis. The students are provided with the opportunity to learn about scientific software, such as SHELX, Olex2, and Mercury, and carry out structure analysis and visual representation of the sponges and liquid molecules. Moreover, hierarchical experiments have been designed to provide flexibility to students and best fit their individual needs and resources. The experiment has been carried out for three semesters in our school. It may refresh students' understanding of crystallography and help them excel in future endeavors especially like synthetic chemistry, pharmaceutical R&D, etc. Credit: *Journal of Chemical Education* (2023). DOI: 10.1021/acs.jchemed.3c00714

The crystal sponge technology is a revolutionary technique, which enables the direct and precise determination of the molecular structure of liquid and gas targets. The technique uses a special network complex to selectively absorb liquid or gas target molecules and order them over long distances, thus achieving a breakthrough in determining the precise structure of liquid or even gas molecules by conventional single-crystal X-ray diffraction technology.

The remarkable technology is recognized as a subversive breakthrough to the traditional single crystal structure analysis technology.

However, due to limitations like experimental reagent toxicity and theoretical knowledge reserve in this work, this technology has not been successfully applied to undergraduate experimental teaching.

Professors from the Chemistry Experimental Teaching Center of the University of Science and Technology of China (USTC) of the Chinese Academy of Sciences (CAS) have designed an overall laboratory experiment with [reaction conditions](#) and characterizations friendly for students to perform and managed to expose undergraduates to the crystalline method, the cutting-edge technique.

The results were [published](#) in the *Journal of Chemical Education*.

Prof. Li Lingling, Prof. Zhu Pingping, and Prof. Zhang Qingwei from the Chemistry Experiment Teaching Center of USTC selected this technology from many cutting-edge research achievements.

In order to enable undergraduates to understand and master this important technology, the team transformed the crystal sponge technology into a safe, universal, and easy-to-use undergraduate teaching experiment for the first time through systematic adjustment and optimization of experimental conditions and overall teaching design and completed 3-semester experimental teaching.

The teaching experiment achieved high-quality training results, winning unanimous recognition from students and teaching supervisors.

The [teaching](#) practice has proved experiment helpful in updating students' understanding of crystallization technology and liquid organic compound structure analysis. It improves [students'](#) innovative thinking and provides strong technical support for their future development in [organic synthesis](#), pharmacology, and other fields.

**More information:** Junxi Zou et al, Capturing the Precise Structure of Liquids: The Crystalline Sponge Method for an Undergraduate Laboratory Course, *Journal of Chemical Education* (2023). [DOI: 10.1021/acs.jchemed.3c00714](#)

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