

## Protocol



# Electrochemical molecular intercalation and exfoliation of solution-processable two-dimensional crystals

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## Abstract

Electrochemical molecular intercalation of layered semiconducting crystals with organic cations followed by ultrasonic exfoliation has proven to be an effective approach to producing a rich family of organic/inorganic hybrid superlattices and high-quality, solution-processable 2D semiconductors. A traditional method for exfoliating 2D crystals relies on the intercalation of inorganic alkali metal cations. The organic cations (e.g., alkyl chain-substituted quaternary ammonium cations) are much larger than their inorganic counterparts, and the bulky molecular structure endows distinct intercalation and exfoliation chemistry, as well as molecular tunability. By using this protocol, many layered 2D crystals (including graphene, black phosphorus and versatile metal chalcogenides) can be electrochemically intercalated with organic quaternary alkylammonium cations. Subsequent solution-phase exfoliation of the intercalated compounds is realized by regular bath sonication for a short period (5–30 min) to produce free-standing, thin 2D nanosheets. It is also possible to graft additional ligands on the nanosheet surface. The thickness of the exfoliated nanosheets can be measured by using atomic force microscopy and Raman spectroscopy. Modifying the chemical structure and geometrical configuration of alkylammonium cations results in different exfoliation behavior and a family of versatile organic/inorganic hybrid superlattices with tunable physical/chemical properties. The whole protocol takes ~6 h for the successful production of stable, ultrathin 2D nanosheet dispersion in solution and another 11 h for depositing thin films and transferring them onto an arbitrary surface. This protocol does not require expertise beyond basic electrochemistry knowledge and conventional colloidal nanocrystal synthesis and processing.

## Key points

- A layered semiconductor crystal that undergoes electrochemical intercalation with alkyl chain-substituted quaternary ammonium cations can be exfoliated into free-standing 2D nanosheets by sonication. The resulting 2D nanosheets in solution are spin-coated to form thin films on versatile substrates for electronic applications.
- The main advantage is the preservation of semiconducting characteristics (e.g., MoS<sub>2</sub>) in the exfoliated nanosheets and assembled thin films.

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# Two Dimensional Crystals

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