Electronic Supplementary Information

Homochirality Beyond Grinding: Deracemizing Chiral Crystals by Temperature Gradient Under Boiling

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Materials and Methods

Reactions under boiling:

Sodium chlorate was purchased from commercial suppliers and used as received. Aqueous suspensions were prepared using bidistilled water. The handedness of the chiral crystals was determined (samples are left to grow for a few hours) by measuring the optical rotation of polarized light with a petrographic microscope (Figure S1).

The protocol described below has been repeated 60 times and in 53% experiments the outcome was dominated by l-crystals, whereas 47% experiments resulted in the opposite enantiomorph.

d-$NaClO_3$ (15 g) and *l*-$NaClO_3$ (15 g) were suspended in water (10 mL) and placed in a 25-mL Erlenmeyer flask. The reaction mixture was heated at reflux, using a 30-cm condenser, in a hot plate at 180-190 ºC. The latter range ensured the optimal thermal gradient to achieve deracemization with almost complete reproducibility; the measured temperature of the mixture at the bottom (thermocouple) was approximately 120 ºC, while the temperature of the supernatant was ~106 ºC. Under these conditions a mass of racemic crystals (~10 g) remained undissolved. After 24 h the reaction mixture was cooled to room temperature and crystals were collected by filtration, and dried under air and vacuum (Figure S2).

Complete deracemization was equally accomplished starting from 10% enantioenriched samples (either *d*- or *l*-$NaClO_3$ as determined by polarizing light) and using the above protocol.

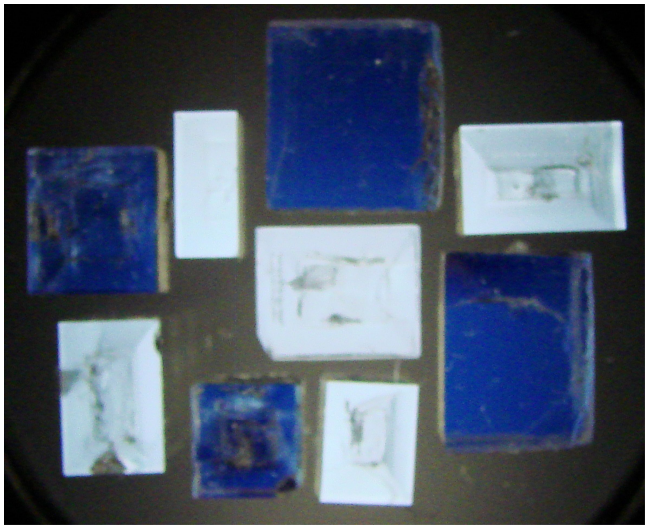

Fig. S1 Photographs of $NaClO_3$ crystals of opposite handedness viewed through cross polarizers.

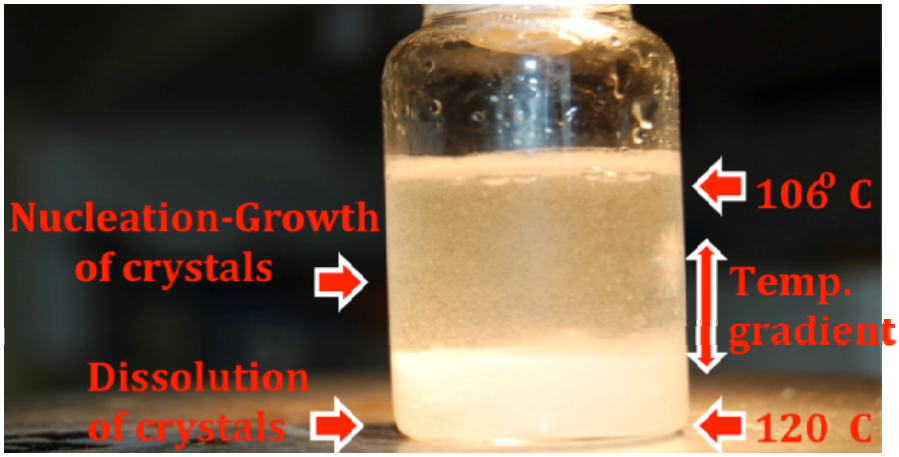

Fig. S2 Photograph illustrating a heterogeneous system under boiling. Crystals dissolve at the hot bottom while the concentrated solution nucleates and grow at the top at a lower temperature. Such cycles comprising dissolution and nucleation-crystal growth give rise to a solid phase of single enantiomorphism in the systems described in the text.

Reactions under boiling and stirring:

The general protocol was applied to racemic mixtures of $NaClO_3$ (by mixing equal amounts of both enantiomorphous crystals as described) and stirring the whole mass at 200 rpm with the magnetic device shown in Figure S3. The latter avoids abrasive grinding of crystals under the horizontal bar. 20 experiments were conducted under such conditions and resulted invariably in a racemic mixture of *d*- and *l*-$NaClO_3$.

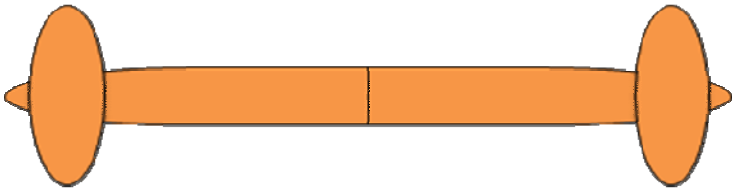

Fig. S3 Home-made Teflon-coated magnetic bar that largely reduces mill effects under agitation and boiling.