

Supplementary Information

Preparation of W/O Nanoemulsion Using Tandem Acoustic Emulsification and Its Novel Utilization as a Medium for Phase-Transfer Catalytic Reaction

Koji Nakabayashi,[†] Hiroyuki Yanagi,[‡] and Mahito Atobe^{†}*

[†]Department of Environment and System Sciences, Yokohama National University, Yokohama 240-8501, Japan

[‡]Department of Electronic Chemistry, Tokyo Institute of Technology, Yokohama 226-8502, Japan

1. Instrumentation

Nuclear magnetic resonance (¹H NMR) spectra were measured on JEOL JNM-AL 400 spectrometer operating at 400 MHz in CDCl₃. All ¹H NMR chemical shifts were reported in ppm relative to internal references of TMS at δ 0.00. EI mass spectra were measured with a Shimadzu GCMS-QP2010 mass spectrometer.

2. Materials

Chloroform (>99%) was purchased from Wako Pure Chemical Industries and used as received. Tetrabutylammonium bromide (>95%), 2-cyanopropionic acid ethyl ester (>95%) and benzyl bromide (>98%) were purchased from Tokyo Chemical Industry and used as received. Potassium carbonate (>99%) and diethyl ether (>99%) and Magnesium sulfate (>98%) were purchased from Kanto Chemical and used as received.

3. Tandem acoustic emulsification

2.0 mL of 50 wt% potassium carbonate aqueous solution was added to 18.0 mL of chloroform solution in glass beaker cell. The 20 kHz ultrasonication to the water/oil mixture was conducted with an ultrasonic stepped horn connected with a 20 kHz oscillator (44 W cm⁻², SONIFIER-250D, Branson Ultrasonics Co.) for 10 min. The sequential ultrasonication with 1.6 MHz treatment after 20 kHz was carried out using an ultrasonic transducer (16 W cm⁻², Honda Electric Co.) connected with a Pyrex glass cylindrical tube (diameter, 24 mm; length, 75 mm) for 10 min. The further sequential ultrasonication with 2.4 MHz treatment after 20 kHz and 1.6 MHz was conducted by an ultrasonic transducer (7 W cm⁻², Honda Electric Co.) connected with a Pyrex glass cylindrical tube (diameter, 24 mm; length, 75 mm) for 10 min. Finally, this procedure

was carried out one more time (two cycles of tandem operation: 20 kHz → 1.6 MHz → 2.4 MHz → 20 kHz → 1.6 MHz → 2.4 MHz).

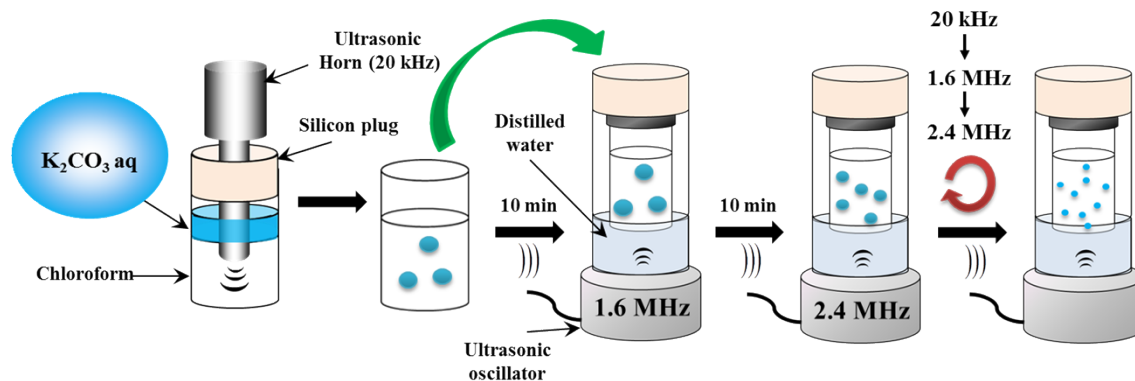
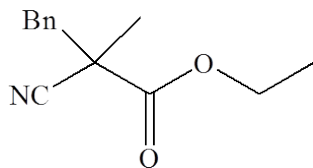


Figure S1. Schematic illustration of the tandem acoustic emulsification treatment of K_2CO_3 aq in chloroform.

4. 3-Phenyl-2-cyano-2-methylpropanoic acid ethyl ester

A solution of 2-cyanopropionic acid ethyl ester (1 mmol), benzyl bromide (1.2 mmol), tetrabutylammonium bromide (0.01 mmol) and potassium hydroxide in chloroform were stirred for 12 hour. Resulting mixture was then extracted with diethyl ether and added with $MgSO_4$. After 30 minute, $MgSO_4$ was removed from solution by filtration. Crude product was purified by silica gel column chromatography to give pale yellow oil (85% yield).



1H -NMR (400 MHz, $CDCl_3$): δ = 7.40 - 7.20 (m, 5H), 4.30 - 4.10 (m, 2H), 3.30 - 3.00 (d, J = 17.5, 10.9 Hz, 2H), 1.6 (s, 3H), 1.2 (t, 3H).

MS (EI, m/z) 217 (M^+), 144, 128, 91, 78.