

Supporting Information

Highly Selective Electrochemical Fluorination of Organosulfur Compounds in Et₃N·3HF Ionic Liquid Under Ultrasonication

Takeshi Sunaga, Mahito Atobe, Shinsuke Inagi, and Toshio Fuchigami*

Department of Electronic Chemistry, Tokyo Institute of Technology, 4259 Nagatsuta, Midori-ku,
Yokohama 226-8502 Japan

E-mail: fuchi@echem.titech.ac.jp

Experimental Section

General Information

¹H NMR (270 MHz) and ¹⁹F NMR (254 MHz) spectra were determined using CDCl₃ as a solvent. The chemical shift for ¹⁹F NMR is given in δ (ppm) upfield from the peak for external trifluoroacetic acid. The product yields were determined by ¹⁹F NMR using monofluorobenzene as an internal standard material. Voltammetry measurements were performed using HOKUTODENKO HABF501. Preparative electrolysis experiments were carried out with Metronnix Corp. (Tokyo) constant current power supply model 5944 by monitoring electricity with HOKUTODENKO Coulomb/Amoperehour meter HF-201. Gas chromatography was carried

out with SHIMADZU GC-8A. Ultrasonication was conducted with HONDA ELECTRONICS Ultrasonic Multi Cleaner W115 for anodic fluorination of **1** and IWAKI GLASS ULTRASONIC CLEANER USC100Z 38s-22 for anodic fluorination of **4**.

Materials

Et₃N·3HF was obtained from Morita Chemical Co. Ltd. (Japan). It is toxic and may cause a serious burn if it comes in contact with unprotected skin. However, proper safety precautions should be taken at all times. It is therefore recommended that rubber gloves should be used. Ethyl α -phenylthioacetate (**1**) and 2-phenyl-3-methyl-4-thiazolidinone (**4**) were purchased and used without purification. The known fluorinated products (**2**, **3**, **5**) were identified by comparison with the spectral data of their authentic samples.^{1,2}

A typical procedure for anodic fluorination

Constant current anodic fluorination of **1** and **4** (1 mmol) was carried out with platinum electrodes (1 x 1 cm²) in Et₃N·3HF (3 mL) in an undivided cell at 24 ± 2 °C. After electrolysis, the yields of the products were estimated by ¹⁹F NMR using monofluorobenzene as an internal standard. After the electrolyte was mixed with 10 ml of water, the product was extracted with ether and dried over anhydrous Na₂SO₄. Then, the extracts were evaporated and almost pure products **2**, **3** and **5** were obtained.

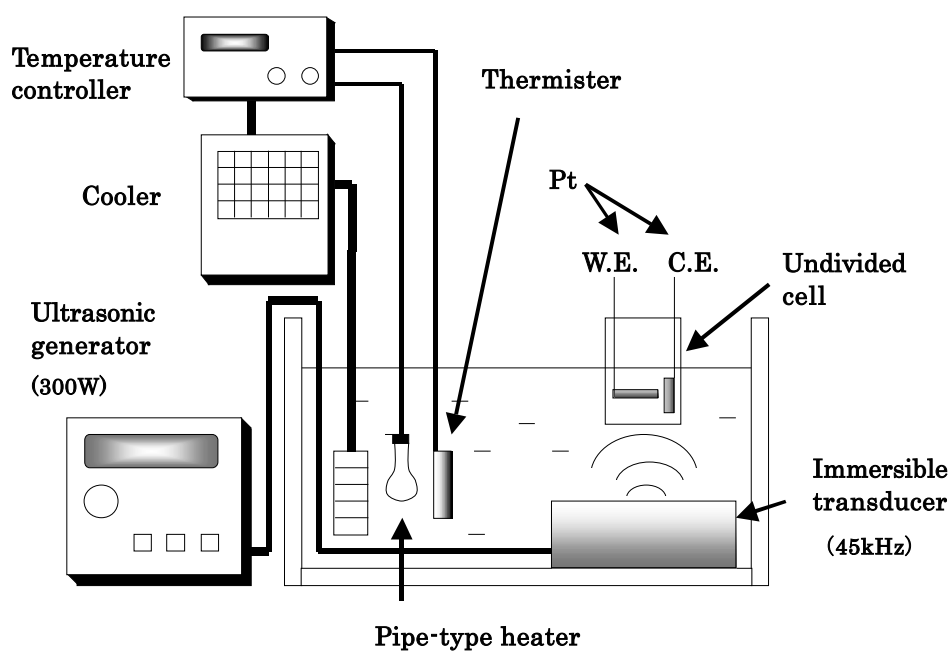


Figure S1. Sonoelectrochemical Reaction System

References

1. T. Fuchigami, M. Shimojo, A. Konno and K. Nakagawa, *J. Org. Chem.*, 1990, **55**, 6074.
2. T. Fuchigami, S. Narizuka and A. Konno, *J. Org. Chem.*, 1992, **57**, 3755.