

## Deprotonation in anodic methoxylation of fluoroethyl phenyl sulfides using site-isolated heterogeneous bases

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### 1. General

<sup>1</sup>H and <sup>19</sup>F NMR spectra were recorded on a JEOL JNM EX-270 (<sup>1</sup>H: 270 MHz; <sup>19</sup>F: 254 MHz) spectrometer in CDCl<sub>3</sub>. The chemical shifts for <sup>1</sup>H and <sup>19</sup>F NMR spectra were given in  $\delta$  (ppm) from internal TMS and monofluorobenzene (-36.5 ppm), respectively. EI mass spectra were recorded on a Shimadzu PARVUM2 gas chromatograph-mass spectrometer.

### 2. Materials

Fluoroethyl phenyl sulfides **1**, **3**, and **5** were synthesized according to the literature.<sup>1</sup> Anhydrous sodium perchlorate, dehydrated acetonitrile, and dehydrated methanol were purchased from Kanto Chemical and used without further purification. 7-Methyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene (MTBD), silica gel supported 1,5,7-triazabicyclo[4.4.0]dec-5-ene (Si-TBD, particle size: 40-63  $\mu\text{m}$ , loading: 0.91 mmol g<sup>-1</sup>), silica gel supported pyridine (Si-pyridine, particle size: 40-63  $\mu\text{m}$ , loading: 1.3 mmol g<sup>-1</sup>), and silica gel supported piperidine (Si-piperidine, particle size: 40-63  $\mu\text{m}$ , loading: 1.1 mmol g<sup>-1</sup>) were purchased from Aldrich.

### 3. Cyclic Voltammetry

Cyclic voltammetry was performed by using a computer-controlled electrochemical analyzer (ALS/CH Instruments 610B). Cyclic voltammetry was carried out with a three-electrode system using a platinum disk ( $\phi = 0.8$  mm) working electrode, a platinum wire counter electrode, and a saturated calomel electrode (SCE) as a reference electrode.

### 4. General procedure for anodic methoxylation of 2,2-difluoroethyl phenyl sulfide

(1)

Anodic methoxylation of **1** (1 mmol) was carried out with platinum plate electrodes (2 x 2 cm<sup>2</sup>) in 0.1 M NaClO<sub>4</sub>/MeOH (10 ml) in the presence of a silica gel supported base using an undivided cell. Constant current electrolysis (5 mA cm<sup>-2</sup>) was conducted with magnetic stirring at room temperature. After the electrolysis, the yield of **2** was calculated by means of <sup>19</sup>F NMR by using a known amount of monofluorobenzene (1 mmol) as an internal standard. The product **2** was identified by comparison with the literature values using <sup>1</sup>H and <sup>19</sup>F NMR and mass spectroscopy.<sup>2</sup>

### References

1. T. Nakai, T. Tanaka, H. Setoi, N. Ishikawa, *Bull. Chem. Soc. Jpn.* **1977**, *50*, 3069-3070.
2. T. Fuchigami, K. Yamamoto, A. Konno, *Tetrahedron*, **1991**, *47*, 625-634.