

Electronic Supplementary Information

Transfer Hydrogenation of Acetophenone in Organic-Aqueous Biphasic System Containing Double Long-chain Surfactants

Experimental

The triphenylphosphinetrisulfonate ($P(m-C_6H_4SO_3Na)_3$, TPPTS) was prepared according to the method described in the literature.¹ Water-soluble ruthenium complex, $RuCl_2(TPPTS)_2$, was synthesized according to the literature.^{2,3} Water, iso-propanol and acetophenone were commercial and degassed before use.

Cetyltrimethylammonium bromide (CTAB), Dihexadecyldimethylammonium bromide ($DDAB_{16}$), sodium dodecyl sulfate (SDS) and polysorbate 80 (Tween 80) were commercial and without further purification. The double long-chain surfactants with different chain length were prepared according to the literature.⁴ The surface tension of surfactant solutions was measured by the Kruss Surface Tensiometer K100.

The transfer hydrogenation was conducted in a Schlenk tube (10ml) with a magnetic stirrer. A typical procedure as follows: the reactor was flushed with argon three times, and then, the ruthenium catalyst, TPPTS, surfactant solution, KOH, water, iso-propanol and acetophenone were added into it respectively. After a given reaction time, the mixture was extracted with chloroform and the organic phase was washed with water, dried with $MgSO_4$ and evaporated to dryness, and then detected by GC.

The composition of product was analyzed on an Agilent 6890 gas chromatograph equipped with a PEG-20M capillary column (30m×0.25mm×0.25mm) and flame ionization detector (FID).

Synthesis of the double long-chain cationic surfactants

1 $C_{16}H_{33}N(CH_3)_2C_2H_5Br$: 3.3 ml (9.81mmol) of $C_{16}H_{33}N(CH_3)_2$ and 1.3 ml (17.4mmol) of C_2H_5Br were performed with 80 ml of acetone as a solvent under argon atmosphere. The reaction was heated to 75°C and refluxed for 24 hours. The resulting products were extracted by ether and further purified by recrystallization from a mixture of acetone and ether. The yield was 3.31 g (89%). mp 208-212°C, 1H NMR($CDCl_3$, 400Hz): δ : 0.881(3H, t), 1.258-1.64(31H, m), 3.405(6H, s), 3.517(2H, t), 3.723(2H, q) ppm.

2 $C_{16}H_{33}N(CH_3)_2C_4H_9Br$: 2.6 ml (7.7mmol) of $C_{16}H_{33}N(CH_3)_2$ and 1.5 ml (13.9mmol) of C_4H_9Br react with 80 ml acetone according to the same procedure as that followed for 1. Overall yield was 1.63 g (52%). mp 58-62°C, 1H NMR($CDCl_3$, 400Hz): δ : 0.897-1.037(6H, t), 1.000-1.698(32H, m), 3.398(6H, s), 3.488(2H, t), 3.527(2H, q) ppm.

3 $C_{16}H_{33}N(CH_3)_2C_8H_{17}Br$: 2.5 ml (7.4mmol) of $C_{16}H_{33}N(CH_3)_2$ and 1.6 ml (9.2mmol) of $C_8H_{17}Br$ react with 80 ml acetone according to the same procedure as that followed for 1. Overall yield was 0.78 g (23%). mp 163-165°C, 1H NMR($CDCl_3$, 400Hz): δ : 0.883(6H, t), 1.231-1.699(40H, m), 3.401(6H, s), 3.535(4H, t) ppm.

4 $C_{16}H_{33}N(CH_3)_2C_{12}H_{25}Br$: 3.1 ml (9.18mmol) of $C_{16}H_{33}N(CH_3)_2$ and 2.5 ml (10.4mmol) of $C_{12}H_{25}Br$ react with 80 ml acetone according to the same procedure as that followed for 1. Overall yield was 2.76 g (58%). mp 173-175°C, 1H NMR($CDCl_3$, 400Hz): δ : 0.897(6H, t), 1.230-1.696(48H, m), 3.396(6H, s), 3.533(4H, t) ppm.

References

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