Transfer hydrodehalogenation of aryl halides accelerated by saturated sodium acetate aqueous solution

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Experimental

Material

Chlorobenzene (99%), fluorobenzene (99%), bromobenzene (99%), iodobenzene (97.5%), benzene (99.5%), acetophenone (98%), aniline (99.5%), 1-propanesulfonic acid sodium salt (99%), Methanesulfonic acid sodium salt (98%), and biphenyl (99.5%) were purchased from J&K Scientific Ltd. 1,2,3-Trichlorobenzene (>98.0%), 1,4-dichlorobenzene (>99.0%), 1,2-dichlorobenzene (>99.0%), 1,3-dichlorobenzene (>98.0%), 4-chlorotoluene (>98.0%), 4-chloroaniline (>99.0%), 3-chloroanisole (>98.0%), 1-phenylethyl alcohol (>98.0%), anisole (>99.0%), and 3'-chloroacetophenone (>97.0%) were provided by TCI (Shanghai) Development Co., Ltd. *n*-dodecane (99%) and sodium iodide (NaI, 99.9%) were obtained from Alfa Aesar. Toluene, sodium formate (HCOONa), sodium acetate (CH_3COONa), sodium sulfate (Na_2SO_4), sodium chloride (NaCl), sodium bromide (NaBr), sodium carbonate (Na_2CO_3) and sodium nitrate ($NaNO_3$), sodium hydroxide (NaOH), dichloromethane (CH_2Cl_2), and sodium propionate (CH_3CH_2COONa) were A. R. grade and were obtained from Sinopharm Chemical Reagent Beijing Co., Ltd. All chemicals were used as received. The catalyst Pd/C with a BET surface area of 326.7 cm^2/g^{-1} (determined by N_2 adsorption-desorption method) was purchased from Baoji Rock Pharmachem Co., Ltd., China (5 wt% Pd, Product No. D5L3), and was characterized by transmission electron microscopy (TEM) as shown in Fig. S1.

Procedure for the transfer hydrodehalogenation of aryl halides

In a typical experiment, desired amount of salts was firstly dissolved in water (5 ml) at desired reaction temperature in a flask of 50 ml heated in an oil bath equipped with magnetic stirring and reflux concentration. Then, HCOONa (10 mmol), sodium hydroxide (1 g), Pd/C (5 wt%, 50 mg), and desired amount of aryl halides were added in the salts solutions. After the reaction was completed, dichloromethane

(CH_2Cl_2, 20 ml) was added to extract the products, and the yield of product was determined through GC (Agilent 6820) equipped with a flame ionization detector (FID) and a HP-5MS capillary column (0.25 mm in diameter, 30 m in length) by using toluene or *n*-dodecane as the internal standard with the following temperature program: hold at 50 ^{o}C for 3 min, 15 ^{o}C/min ramp to 260 ^{o}C and a final hold for 3 min. In addition, identification of the products was also done by GC-MS (Shimadzu QP2010).

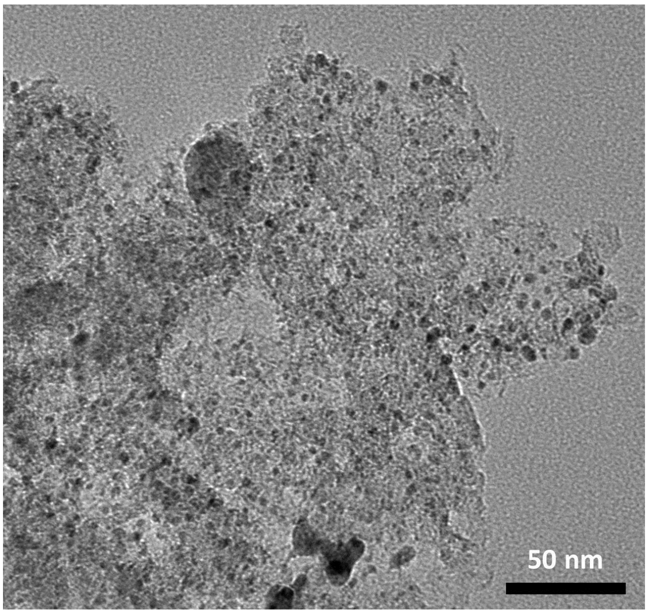

Fig. S1. TEM image of the commercial Pd/C catalyst.