The Calculation of Quaternary Ammonium Salt Yield

The calculation of quaternary ammonium salt yields is based on ^{1}H NMR analysis Taking tetramethyl ammonium bromide as an example, we did it according to the following method:

As shown in Fig. 1a and 1b, ^{1}H NMR spectrum of a sample obtained from the reaction of ammonium bromide with dimethyl carbonate in the presence of 1-ethyl-3-methylimidazolium bromide (EMImBr) and a standard tetramethyl ammonium bromide sample, respectively. In Fig. 1a, peak (a) at chemical shift 1.42 ppm is assigned to the hydrogen on the methyl group of the ethyl group on EMImBr, and peak (b) at chemical shift 3.12 ppm (the same as that of standard tetramethyl-ammonium in Fig. 1b) is assigned to the hydrogen atoms of the methyl groups of tetramethylammonium. Since that EMImBr acts as a catalyst, it's amount (M_{IL}) should keep constant before and after reaction. The amount of tetramethylammonium (M_{TC}) thus can be calculated based on the peak areas of hydrogen at chemical shift 1.42 ppm (A_H^a) and that at chemical shift 3.12 ppm (A_H^b) in Fig.1a according to the equation (1). All of the quaternary ammonium salt yields can be calculated according to similar method.

$$M_{TC}=M_{IL}\,A_H^b/(4\,A_H^a) \qquad (1)$$

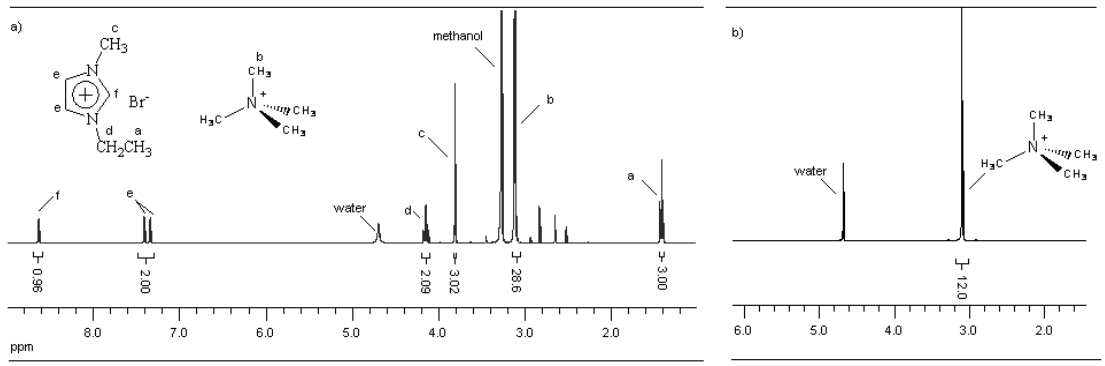

Fig. 1. The ^{1}H-NMR spectrum: a) sample after reaction of ammonium bromide with dimethyl carbonate in the presence of BMImBr catalyst, b) standard tetramethylammonium bromide sample.

Isolation

In our investigation, our product quaternary ammonium salts can be separated by direct filtration and wash by acetone, such as tetramethyl ammonium salts, which does not dissolve in acetone. More than 92.0% of the product can be recovered. Of course, for long chain involved quaternary ammonium salts, because of the high solubility quaternary ammonium salts in acetone, the recovering ratio is low. Generally, the products can be separated by recrystallization in a suitable solvent. This is a long-run research work. The investigation of this work is still in progressing.