Extraction method

Protocol (liquid-liquid)

- Separatory funnel
- Mixing and release of pressure
- Separated

About 3 times

- Halogenated solvent will be the lower layer.
- Organic layer
- Aqueous layer

The obscure interface is checked by adding water or NMR tube and lighting.

NMR tube
Extraction method

Protocol (liquid-liquid, small scale)

1. To check completion of the extraction, analyze the aqueous layer by TLC.
2. To extract highly polar products, use $\text{CH}_2\text{Cl}_2:\text{MeOH} (9:1)$ or THF after adding brine to the aqueous layer or evaporate the aqueous layer.
3. To avoid emulsions, gently shake and swirl the funnel.
4. To destroy emulsions, add brine or hexane to the mixture or filter out impurities by Celite.
5. To remove DMSO or DMF, use hexane/EtOAc (3/1) or $\text{Et}_2\text{O}$. 

Tips

- Vortex mixer is also applicable.
Extraction method

6. To remove less-polar byproducts, firstly dissolve a product to aqueous layer and then to organic layer again by adjusting the pH of aqueous layer.

Ex.) Back extraction of carboxylic acid

Less-polar byproducts are removed.
The organic solvent should

1. Dissolve a product to be extracted.
2. Not react with a product to be extracted.
3. Not react with or be miscible with water.
4. Have a low boiling point.
Dehydration method

Protocol

Ex.) Data of drying agent in descending order of drying capacity

\[
\text{Na}_2\text{SO}_4 \ (1.2 \text{ g H}_2\text{O/g dessicant}) > \text{MgSO}_4 \ (0.2-0.8 \text{ g H}_2\text{O/g dessicant}) \geq \text{CaCl}_2 \ (0.2 \text{ g H}_2\text{O/g dessicant})
\]

http://www.nacalai.co.jp/information/trivia2/01.html