

Some Common Solvent Purification Methods

References:

1. Riddick, J. A.; Bunger, W. B.; Sakano, T. K. (1986) Organic Solvents (4th ed.), John Wiley & Sons, New York.
2. Gordon, A. J. and Ford, R. A. (1972) The Chemist's Companion, John Wiley & Sons, New York, pp 431-436.

I have also included a few procedures which I have found to be superior to methods suggested by the above references.

Acetone (BP 56.2)

Dry over Molec sieves or K_2CO_3 . Removal of aldehydes by $KMnO_4$. High purity: saturate w/ dry NaI, then chill to -10 C, filter off NaI crystals. Distill over sieves.

Acetonitrile (BP 81.6)

Predry if wet, reflux & stir w/ CaH until gas evolution ceases. Distill and store over sieves.

Benzene (BP 80.1)

Stir w/ H_2SO_4 (.1l/l benzene) and separate acid. Repeat until no darkening occurs, then distill.

Chloroform (BP 61.2)

Shake w/ con H_2SO_4 , wash w/ water, dry and distill from K_2CO_3 . Alternatively, pass through column of Grade I activated alumina (50g/L solvent).

Diethyl ether (BP 34.5)

Check for peroxides. Pretreat with Na wire, then add LAH (or CaH) and distill.

Diglyme (BP 161)

Heat over Na, then distill.

DMF (BP 152)

Stir w/ KOH, filter and distill from CaO or BaO.

DMSO (BP 189)

Dry overnight w/ Drierite, BaO, CaO, or NaOH. Distill from BaO, CaO, or NaOH (2-3mm, 50 C).

p-Dioxane (BP 102)

Reflux over Na until the metal remains shiny then distill, store in dark under N₂. To remove acetal impurities, reflux a mixture of 300 ml H₂O, 40 ml HCl and 10 ml p-dioxane for 12 h under N₂. Cool, add KOH until no more dissolves, then decant the dioxane layer and dry w/ KOH. Distill as above.

DO NOT USE LAH!!!!

Ethanol (BP 78.3)

For anhydrous from abs: Reflux 60 ml EtOH, 5 g Mg, and a few drops of CHCl₃ or EtBr (catalyst) until all Mg converted to the oxide. Add 900 ml EtO then distill, store over sieves. Abs EtOH can be prepared from 95% if benzene must be excluded (no denaturants if USP grade) by refluxing over CaO for 24 hours, then distilling.

Ethyl Acetate (BP 77.1)

Wash with 5% Aq Na₂CO₃, then sat. CaCl₂; dry over K₂CO₃ and distill from P₂O₅.

Hexane (BP 68.7) See *Saturated Hydrocarbons***Methanol (BP 64.5)**

Most water removed by storage over CaO then distillation from CaO. Storage over sieves (NO DRIERITE!!). Ultrapure: 50 ml MeOH, 5 g Mg and 0.5

until I₂ color disappears, then add 1 L MeOH and return to reflux 1 h. Distill slowly.

Methylene Chloride (BP 40.8)

Wash with con H₂SO₄, sat. Na₂CO₃, and water. Dry over CaCl₂ and distill from P₂O₅. Alternatively, distillation from anhydrous K₂CO₃ and storage over sieves gives pretty good product.

Pyridine (BP 115.3)

Dry over KOH for ~24 h, then distill from BaO or CaO. Store over sieves.

Saturated Hydrocarbons

Most contain small amounts of olefin/aromatic. For removal, shake with a mixture of H₂SO₄ and HNO₃ 3-4 times, wash water, dry CaCl₂, and distill.

Tetrahydrofuran (BP 66)

Store over iron powder to inhibit peroxide formation, filter before purification. Remove peroxides by refluxing with 0.5% (w/v) CuCl for 30 min., then reflux from KOH, reflux over Na for several hours, then cool to ~40° and add benzophenone; distill when deep blue color is obtained.

Toluene (BP 110.6)

Dry over CaCl₂ (only if very wet) then distill.

The preceding information is intended for use by experienced chemists only. If you are not familiar with the reagents listed, do not attempt to use this information.

