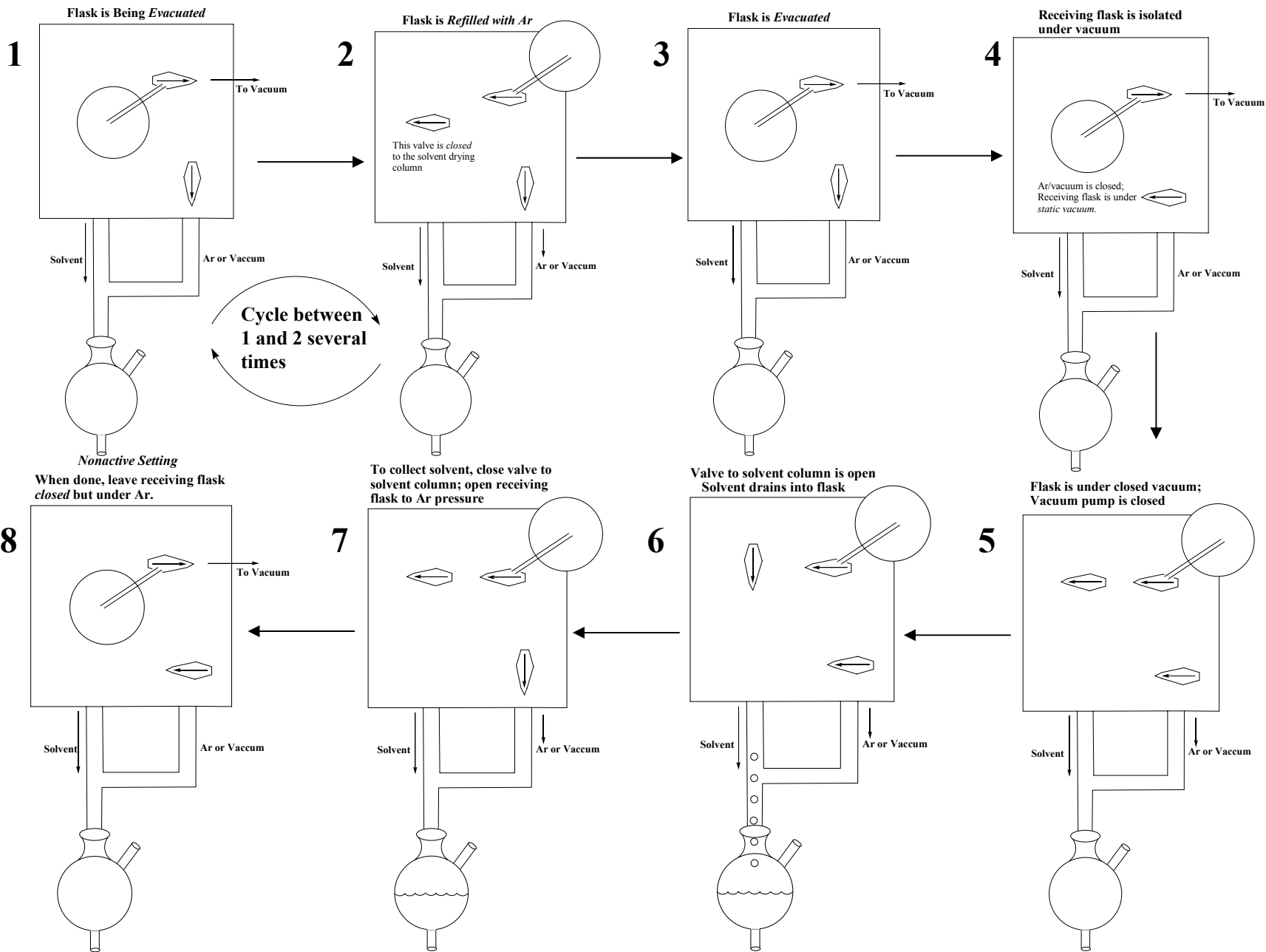


Graphical Guideline to Collecting Solvents with the Glass Contour System



Procedures for Killing and Regenerating of Non-Na/K Stills

	Solvent	Drying Agent	Killing	Regenerating
1.	Methanol	Mg metal	A	A
2.	Ethanol	Mg metal	A	A
3.	Acetonitrile	CaH ₂	B	E
4.	CHCl ₃	P ₂ O ₅	A	B
5.	CH ₂ Cl ₂	P ₂ O ₅	A	B
6.	CCl ₄	P ₂ O ₅	A	B
7.	Acetone	K ₂ CO ₃	A	D
8.	Propanol	Mg metal	A	A
9.	Pyridine	CaH ₂	B	C

Killing Procedures

Procedure A. Turn off nitrogen. Let the flask cool. Carefully remove flask and carry it to the hood. Put it under N₂. Slowly add H₂O (15 mL). Stir and let stand for 15 minutes. If cool enough, slowly add 50 mL more H₂O while stirring. Discard appropriately. Clean and oven-dry the flask (also distillation apparatus, if necessary).

Note: If at any time the solution boils vigorously during this procedure, stop adding water until it is cool again.

Procedure B. Turn off nitrogen. Let the flask cool. Carefully remove the flask. Stopper it lightly and carry it to the hood. Slowly add 10 ml of isopropanol or *t*-butanol. When cool, add 20 mL more. Stir and when cool add 50 mL of a 2:1 ethanol:water solution. Let stand 1 hour. Add 20 mL water. Let stand 1 hour. Discard as appropriate.

Regenerating Procedures

All apparatus (predried in the oven) should be assembled while hot and flushed with N₂.

Procedure A .

Methanol – 50 mL methanol, 5 g Mg turnings (dried overnight at 105 °C), and 0.5 g freshly sublimed I₂ are added. Stopper the 2-necked flask and reflux gently until the I₂ color disappears. Be careful not to overheat as H₂(g) is vigorously evolved. The mixture will be grayish-white. Cool the flask and add more solvent. Vigorously stir and reflux for 24 hours.

Ethanol – same as above except: a) Use 60 mL absolute ethanol. b) Use a few drops of CCl₄ or CHCl₃ (better than I₂).

Procedure B. Add solvent to the flask and add 6 scoops of P₂O₅ powder into this flask. Reflux 24 hours prior to using newly regenerated.

Procedure C. Predry pyridine over KOH. Transfer pyridine to flask. Add 3 scoops of CaH₂ to the solvent. Reflux 48 hours prior to use. Discard the first 50 mL.

Procedure D. Same as B, but use K₂CO₃ instead of P₂O₅.

Procedure E. Dry ~100 g of alumina gel (Neutral, 80 – 200 mesh) in an oven overnight @ > 120 °C. Transfer the alumina gel to an appropriately sized column and allow to cool. Run predried CH₃CN to the still. Add 5 scoops CaH₂ to the flask and reflux for 24 hours before

References;

Solvents 1-6, 8, 9: *Chemists Companion*, Arnold J Gordon Richard A. Ford, John Wiley and Sons, NY, 1972.

Solvent 7: Calderazzo, et al *Inorg Chem.* **1983**, 22, 1865.