

## Maintenance of the Solvent Stills

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This job consists of taking care of the distillation of common solvents for the group. Under the large hoods of both labs, you can find the stills for the following solvents:

Acetonitrile ( $\text{CH}_3\text{CN}$ )  
Benzene (PhH)  
Dichloromethane ( $\text{CH}_2\text{Cl}_2$ )  
Diisopropylethylamine (Hünig's base)  
Ether ( $\text{Et}_2\text{O}$ )  
Hexanes (Hx)  
Tetrahydrofuran (THF)  
Toluene (PhMe)  
Triethylamine (TEA)

### Use

To distill and obtain the solvents, you have to follow a general simple procedure that tells you how to handle syringes, needles and tags. Make sure you ask any senior student about it before attempting anything. The variac settings and names of the solvents are labeled at the top of the hood.

Since it uses the combination of heat and highly flammable solvents, this is the most dangerous area of our labs (consult the Laboratory Safety Section from Tour's Handbook). It is everybody's responsibility to be aware of potential hazards. Be alert and use common sense. Never (!) leave a still unattended for extended period of time (this includes leaving for group meeting). Feel free to inspect the distill area, but only I have the authority to make modifications of apparati, settings and additions of solvents and reagents.

Verify that safety points of the stills:

- Level of N<sub>2</sub> flow and solvents
- Variac's settings and function
- Connections, fuses and cables
- Valves, stopcocks and septas
- Oil bubblers
- Possible leaks and cracked glassware
- Color of the solvents.

If you have a special need (e.g., an extra large amount of solvent) or if you have any concern about the stills do not hesitate to let me know immediately.

The following responsibilities are also included in this job:

- Maintenance of the glassware, cooling and N<sub>2</sub> systems and other apparatus.
- Keeping a good stock of the drying, reagents and solvents.
- Following proper drying methods, see chart below.
- Any other associated designations that may be required. Further information about the use and maintenance of these areas can be found in the Perrin's Purification of Laboratory Chemicals. Dr. Tour and Jake have a copy of this book.

<b>Solvent</b>	FW	D g/mL	Bp (°C)	Polarity Index	Refractive Index (25°)	Viscosity (cP, 25°C)	Drier agent
CH <sub>3</sub> CN	41.05	0.786	82	6.20	1.341	0.34	CaH <sub>2</sub>
PhH	78.11	0.879	80	3.00	1.498	0.60	CaH <sub>2</sub>
CH <sub>2</sub> Cl <sub>2</sub>	98.96	1.327	40	3.40	1.421	0.41	Boiling stones
Hünig's base	129.24	0.738	127		1.41	NA	CaH <sub>2</sub>
Et <sub>2</sub> O	74.12	0.708	35	2.90	1.350	0.24	Sodium benzophenone ketyl
Hx	100.20	0.659	69	0.20	1.385	0.30	Boiling stones
THF	72.11	0.886	67	4.20	1.405	0.46	Sodium benzophenone ketyl
PhMe	92.14	0.867	111	2.40	1.494	0.55	CaH <sub>2</sub>
TEA	101.19	0.726	88.8		1.400	NA	CaH <sub>2</sub>