Solvents: Distill On-Site or Buy Pure?

Two Purification Methods

Most organic chemistry laboratories require extremely pure solvents with no moisture or oxidation. One way to purify these solvents is to distill them on demand. However, distillation on-site requires significant energy, cooling water, supervision and maintenance. The process also requires a fume hood, condensers and flasks, heating mantles and safety switches. Shipping the solvent cans or bottles also generates waste in the form of packing materials (metal cans, cardboard, Styrofoam etc.). Finally, the process is relatively dangerous as accidents have been known to lead to explosions.

An alternative to distillation is buying purified and flushed solvents with dispensers that scrub water or oxygen. Some vendors such as EMD Chemicals Inc. will deliver solvents through their EM ReCycler® program[1] in stainless steel containers. When the containers are empty, EMD replaces them with full ones and returns the empty to their facility to be cleaned and refilled. This program has some advantages. It is safer for lab personnel than stills because the solvents are not being heated on-site, and they eliminate waste associated with transporting and distilling solvent. The containers also take up significantly less space than a distillation system and do not require a dedicated fume hood. The containers are sealed to avoid contamination by air and water. Finally, the ReCycler program can be very convenient for busy laboratory groups, freeing up time for long synthesis procedures. EMD sells adapters that dispense solvents safely and cleanly. Other vendors such as Innovative Technologies supply molecular sieve, silica gel purifiers and argon flushing apparatus.

LabRATS staff and a graduate student volunteer made a cost comparison for these two systems. We assumed that high volumes of three solvents (60-80L per year) were needed, while low volumes (5-10 L) of three more solvents were also needed by this hypothetical lab. We did not make estimates of labor differences.

Mentioning of brand names does not indicate endorsement of these products.

[1] http://www.emdchemicals.com

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On-Site Solvent Distillation

We analyzed costs required for distilling solvents for one year, including one-time purchases. Costs per year assume the same volume for each solvent as with purified canisters. UCSB has a strategic contract with Fisher Scientific and the prices below are current as of summer, 2007 for ACS grade solvents. Because there is no minimum purchase of bottles, the costs per liter are maxima.

Solvent	Cost/Container	Cost/Year	Cost/Liter
MeCN (20L)	$154	$496	$7.70
DCM (4x4L)	$74	$592	$7.40
THF (20L)	$176	$704	$8.80

Solvent cost for 1 year: $1758

Additional recurring chemical costs:

Chemical	Size	Cost
CaH	100 g	$250
Na	100 g in mineral oil	$55
Benzophenone	500 g	$20

Reagent Costs for 1 year: $325

Set-up costs for solvent stills:

Part	Size	Cost
Heating Mantle + Controller	1L	$262
Round Bottom Flask	1 L, 24/40 joint	$30
Reflux Condenser	200 mm, 24/40 joint	$120
Solvent Collection Bulb	1 L, 24/40 joints	$300
Timer and power shut-off	Fabricated on-campus	$300 (?)
Manifold (1 ea)	4 outlets, all glass	$220

Power cut-off switches or timers are needed to make sure the stills don't boil dry, and the power cut-off should include a pressure sensor in cooling water fails. Be sure to include water shut-off when power is cut off by the timed switch. This will save thousands of gallons of water if it is not turned off assiduously. Assuming three stills,

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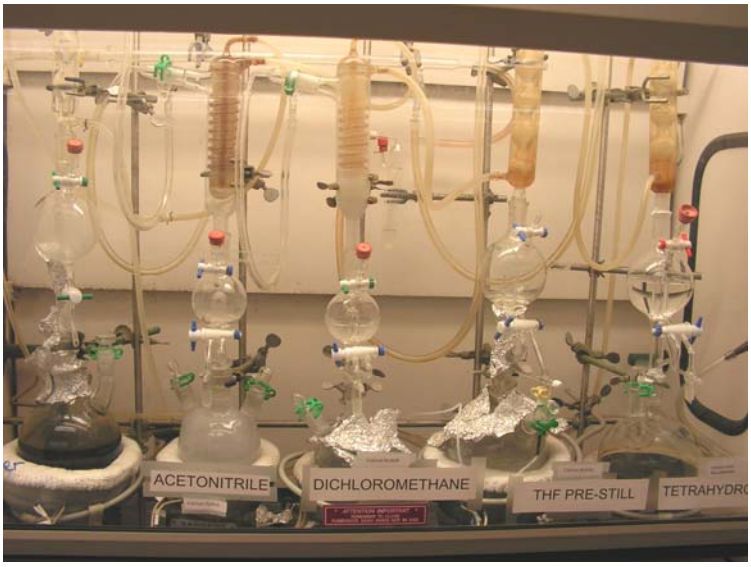
total cost for glassware and heaters: $2566. The energy cost of one VAV hood is <$500 if the sash is kept closed when not attended.

Inert gasses:
$N_{2(g)}$: One tank: $8.64, at one tank/mo: $103.68/year
$Ar_{(g)}$: One tank: $25, at one tank/mo: $300/year

Total cost for on-site distillation of solvents:
First year: $5,053
Following years (First year's cost minus glassware costs): $2,487



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Delivery of purified solvents

Shown below is a cost analysis of the ReCycler program with some of the most common organic solvents. Other solvents, such as diethyl ether and ethyl acetate are available. Costs shown are for 18.9 L containers. One disadvantage is that there is a minimum annual charge. If smaller volumes than the minima are used, then the cost per liter rises substantially. This service is not available for small volumes, less than 60 L per year.

Solvent	Containers/ Year, Liters	Containers in use	Cost/ Container	Cost/ Year	Cost/ Liter
MeCN	3, 57	2	$550	$1,650	$29.10
DCM	4, 76	2	$550	$2,200	$29.10
THF	4, 76	2	$550	$2,200	$29.10

MeCN= acetonitrile; DCM = Dichloromethane; THF = Tetrahydrofuran.Total cost for 1 year: $6,050

The customer is charged only when a full container of solvent is delivered. However, these costs are minima, as the number of solvent containers/year shown above is a minimum

Additional costs:
Solvent dispensing head: $1,689
For three containers: $5,067
The dispensing heads are a requirement of the program, and would only have to be purchased once.

Total cost for EM ReCycler program:
First year: $11,117
Following years: $6,050

Conclusions

The start-up costs for the EM ReCycler program are ~$6,000 higher than those for distilling solvents onsite. This is due to the cost of the solvent dispensing heads required for the EM ReCycler containers. Assuming they do not need to be replaced, the EM ReCycler program is also >$3,000 more expensive per year than on-site distillation for following years.

The costs above do not include those associated with a dedicated hood. Including air cooling or heating energy drawn up by the hood blowers, there is an additional cost of $1,000-3,000 per year to the university which is not borne by the laboratory. If the hood sash is kept closed these energy costs drop dramatically. In the Chemistry Department hoods are “smart” or variable air volume (VAV) depending on sash position.

It appears that distillation on-site saves several thousands of dollars in start-up and annual costs. With installation of power and water cut-off timers this method can be relatively safe and reduce energy consumption, water and chemical use.

On the other hand the loss of a hood in a research laboratory can eliminate two or three graduate student workers. As well, any increases in hazards are also a high cost that many researchers avoid. Thus many organic chemists at UCSB prefer to set up their labs with regular delivery of purified solvents that can be purged and polished on-site without distillation.