

Solvent Purification System (SPS)

Aaron, Rob

Updated July 12, 2022



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Solvent Purification System

Solvents dispensed are:

1. Dry – passed over drying columns
2. Low O₂ content* – sparged with and stored under argon
3. Unstabilized – free of radical inhibitors

* For highly air-sensitive reactions freeze-pump-thaw cycle is recommended.



Solvent taps in right glove box

Drying Column

Solvent Purification System



Solvent	12 Month Usage
THF (yellow)	18.0 L
Diethyl ether (red)	4.6 L
DCM (blue)	42.0 L
Toluene (black)	8.7 L
Hexane (green)	2.7 L

Solvent Kegs

- For refills ask one of the persons in charge
- If you suspect the kegs are empty, you can lift the keg slightly to see if there is solvent left
- SPS solvents are to be restocked immediately after refill



SPS Solvent Cabinet

(Instrument Room)

Ask Aaron or Rob for refills

Do not use SPS solvent for **any other purpose.**

Do not store **any other solvent** in this cabinet



Argon

- Check if cylinder is empty before use (at or below **500 psi**). **Let us know!**
- Pressure is set to **10-12 psi**. **Do not change**
- Pressure is high – use a flask that prevents splashing of the solvent
- Only Ultra High Purity Argon can be used



Operation

- Bring a dried **suitable flask** (molsieves optional)
- Do not take much more solvent than needed. **Avoid unnecessary storage** of solvents in lab, especially ethers
- Follow the printed instructions. If you are unsure or need training, **just ask Aaron or Rob**



Solvent System Dispensal

Read and understand dispensal protocol before using the system!
Misuse will result in revocation of solvent system privileges!
When the flask is under argon, be sure to **HOLD** it!

(Receptacle is under *vacuum*)

1. Turn A to left.

(Receptacle is now under *argon*)

2. Remove receptacle and place a **properly dried flask** on the system.

(Flask will be filled with *argon*)

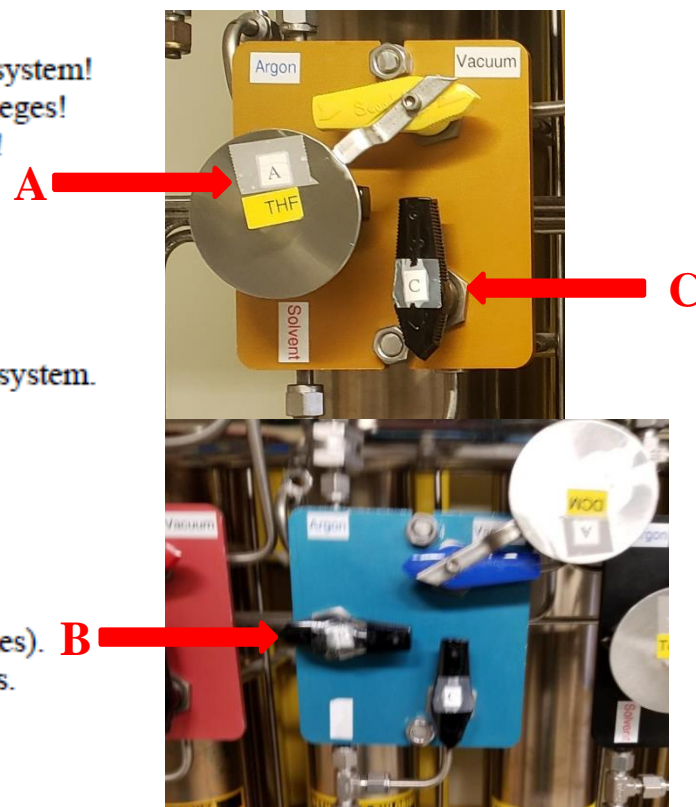
3. Turn A to right.

(System is now under *vacuum*)

4. Backflush the flask 3-4 times with *argon*.
-Turn A to left, pause, then turn A to right (repeat 2-3 times).
5. Allow the flask to remain under *vacuum* until the pump stops.
6. Turn C to left.
7. Turn A to left.
8. Turn B down.


(Vacuum draws solvent into the flask)

*Warning: Solvent fills very quickly!



Removing Schlenk or other non-Strauss flasks

*Warning: Solvent fills very quickly!

9. Turn B to left to stop solvent.
10. Turn C down.  **Solvent can Splash**
(Purges solvent in the line)
11. Remove flask.
(System should be expelling *argon* after the flask is removed)
12. Wipe tip with kim wipe to remove any residual solvent.
13. Return receptacle to system.
14. Turn A to right.
15. Fill out log book.

After Step 10 your flask is under argon pressure.

Turn valve C to the left to remove the flask.

Otherwise the solvent can splash **if not using a Strauss flask**



What if you accidentally sucked solvent (vapor) in the pump? -> **Flush the Pump**



No cap, pump pulls air for 10 minutes



After 10 min replace cap



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March 31, 2022

******IMPORTANT ANNOUNCEMENT FOR CUSTOMERS OF
JC MEYER SOLVENT SYSTEMS******

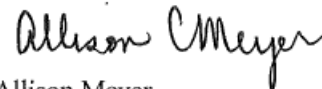
Dear Friends:

I have now completed my due diligence investigation for a replacement solvent system provider. As you recall, my husband, Jorg Meyer, passed away last May 2021. The provider I have selected is CHEMBLY, located at ICT SKVI B-dong 610-ho, 58, Giheung-ro, Giheung-gu, Yongin-si, Gyeonggi-do, Republic of Korea, with e-mail: sales@chembly.com, tel: 82-2-6952-1200 (office)/ 82-10-8791-3910 (mobile). The owner of Chembly is Chan Jin Park. CHEMBLY has representatives located in many countries, and a representative in the United States, Brian Choe, who is located in Irvine, California, email: brian@packaid.com, tel. (562) 843-8443 (mobile).

JC Meyer Solvent Systems ("JCM") has provided all of its trade secrets, instructions, specifications, drawings, computer assisted drawings, inventory, and pertinent records to CHEMBLY. I am confident that CHEMBLY will be able to fulfill all of your needs for maintenance/repair of your existing systems and installation of new systems.

I anticipate that Chan Jin Park or Brian Choe will be contacting you soon. Please explain your current needs and how CHEMBLY can assist you in meeting those needs. Thank you again for the many years of allowing JCM to be of service to you.

Respectfully yours,



Allison Meyer

cc: Chan Jin Park
sales@chembly.com





Thank you!




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Cite this: DOI: 10.1039/d0dt02345k

Evaluation of air-free glassware using the ketyl test†

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Air-free glassware and techniques are used in most organometallic research laboratories, but their relative effectiveness has not been well studied. We report a method for quantitatively comparing air and moisture exclusion by air-free glassware. Samples of benzophenone dianion in various glassware are monitored by video as their purple color is quenched. Numerical values for time until colorless are extracted from the video data and interpreted as a relative measure of air and moisture exclusion. Low pressure/vacuum (LPV) NMR tubes demonstrated by far the best performance, compared to a variety of Schlenk flasks and standard NMR tubes. The findings are of immediate practical use for synthetic chemists, and the evaluation methods can be used by individual laboratories to assess other conditions for air and moisture exclusion.

Received 3rd July 2020,
Accepted 27th July 2020

DOI: 10.1039/d0dt02345k

rsc.li/dalton

by Texas A & M University on 10/5/2020 9:16:22 PM.

Table 1 Normalized mean ketyl test time until colorless for different types of glassware

Glassware style	Time until colorless ^a (min)
25 mL Schlenk tube, greased ground glass stopper (A)	300 ± 10
50 mL Schlenk tube, greased ground glass stopper (B)	277 ± 46
50 mL round-bottom Schlenk flask, greased ground glass stopper (C)	129 ± 27
25 mL Schlenk tube, threaded PTFE stopper (D)	391 ± 57
NMR tube, standard cap (E)	413 ± 106
LPV NMR tube, threaded PTFE stopper (F)	5200 ± 400

^a Times are reported with 95% confidence intervals. Times were normalized to 300 min for concurrently monitored standard 25 mL Schlenk tubes with ground glass stopcocks and stoppers. The number of trials was 65 for Schlenk tubes A, ten for NMR tubes E, and nine for all other glassware.