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Protocol for Drying Deuterated Solvents - Burns Research Group - Spring 2010

Procedure #1

<u>Solvent</u>	<u>Agent</u>
CD ₂ Cl ₂	CaH ₂
C ₆ D ₅ Cl	CaH ₂
C ₆ D ₅ Br	CaH ₂
CDCl ₃	CaH ₂
Cl(CD ₂) ₂ Cl	CaH ₂
Cl ₂ (CD) ₂ Cl	CaH ₂

Lower Boiling Solvents

- 1) Place CaH₂ in a Schlenk flask (with a stirbar) in a glove box. CaH₂ should be a grey, free-flowing powder/solid. Cap Schlenk flask with septum
- 2) Outside glove box, pour solvent over CaH₂. Place solvent under a static N₂ pressure. Stir for for 1-2 days. The longer the better.
- 3) Attach a 90° Adapter to Schlenk flask w/CaH₂ and solvent under N₂.
- 4) Freeze-pump-thaw solvent 3 times.

Freeze-Pump-Thaw: Place liquid N₂ bath around flask. Once solvent is frozen solid open vessel to vacuum. Evacuate flask. Once flask is under full vacuum, seal flask and warm to room temperature. (This is known as a Freeze-Pump-Thaw procedure.)

- 4) Vacuum transfer degassed solvent into a dried, evacuated storage container. Label the container.

Procedure #2

<u>Solvent</u>	<u>Agent</u>
Benzene- d_6	Na / Benzophenone
Toluene- d_8	Na / Benzophenone
THF- d_8	Na / Benzophenone

- 1) In a glove box, place Na/benzophenone in round bottom flask with stir bar and attach 90° adapter. **Note:** For benzene- d_6 and toluene- d_8 add the solvent to the Na/benzophenone in the glove box.
- 2) Attach drying set-up to line and evacuate. Once flask is evacuated seal flask from dynamic vacuum.
- 3) In a separate Schlenk vessel, add desired amount of THF- d_8 . Attach vessel to line. Perform a Freeze-Pump-Thaw procedure on THF- d_8 (See step 3 procedure #1 for full description). **Note:** For benzene- d_6 and toluene- d_8 simply perform Freeze-Pump-Thaw procedure on solvent/Na-benzophenone mixture three times.
- 4) Repeat Freeze-Pump-Thaw procedure on THF- d_8 two more times.
- 5) Vacuum transfer degassed THF- d_8 to flask containing Na / Benzophenone. Warm solvent to room temperature and stir under a static vacuum for several hours.
- 6) Attach hot Schlenk solvent storage flask to line and evacuate. Let flask cool to room temperature under a dynamic vacuum. Once at room temp. close flask to vacuum.
- 7) Vacuum transfer dried solvent from Na / benzophenone to empty, dried Schlenk solvent storage flask. Label flask.

Procedure #3

<u>Solvent</u>	<u>Agent #1</u>	<u>Agent #2</u>
Acetone - d_6	4 Angstrom Sieves	4 Angstrom Sieves
DMSO - d_6	CaH ₂	4 Angstrom Sieves

<u>Solvent</u>	<u>Agent #1</u>	<u>Agent #2</u>	<u>Agent #3</u>
CD ₃ CN	CaH ₂	4 Angstrom Sieves	4 Angstrom Sieves

- 1) In a glove box, place drying agent #1 in round bottom flask with stir bar and attach 90° adapter.
- 2) Remove drying set-up from box and quickly pour desired amount of solvent into round bottom flask.
- 3) Attach flask to line and perform Freeze-Pump-Thaw procedure on solvent three times. Warm flask to room temperature and let solvent stir for 12 hours under static vacuum.
- 4) In the case of DMSO - d_6 , after stirring for 12 hours, perform two more Freeze-Pump-Thaw procedures on the solvent.
- 5) Bring a Schlenk solvent storage flask into the box and place drying agent #2 (sieves) in the flask.
- 6) Attach the Schlenk solvent storage flask to line and evacuate it.
- 7) Vacuum transfer solvent from round bottom flask with drying agent #1 to Schlenk solvent storage flask. Both of these solvents are stored over sieves while in use. Label flask.
Note: For CH₃CN, repeat step 5-7 using drying agent #3. Store CH₃CN over sieves.