

SOP: Distillation of toluene over calcium hydride

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1. **Anyone using the distillation room for the first time must receive training** from the responsible person (see above) **and read all relevant SOPs.**
2. Use the booking sheet to book time in advance.
3. Fill in the 'use of solvent stills' form so everyone knows who's currently using the still.
4. Make sure the water to the condensers is on (feel the condenser, it should be cold to the touch). **Never turn off the water to the condenser.**
5. Do a visual inspection of the set-up to make sure there are no cracks or other irregularities on the still. **If anything is broken or out of order, please contact the responsible person.**
6. Make sure heating mantle is off and unplugged before filling.
7. Remove the clip and stopper from one of the necks of the round bottom flask and fill with toluene using a plastic funnel. **Never fill above the mark on the flask. Bring your own solvents.** Add a large spoon of **calcium hydride**. Add fresh boiling stones as needed. Put the stopper back on the flask and make sure the clips are secure.
8. Insert the **plug into the digital timer**, turn on the switch at the base of the mantle and turn the **dial to 6**. The digital timer must be used; it runs from 8am to 6pm on weekdays and is there as a safety measure to prevent the stills from being left on overnight.
9. Turn the stopcock on the collection bulb (labelled 1 in the picture) to a position where all the outlets are closed.
10. For emptying the collection bulb, **turn off and unplug** the heating mantle and put a bottle on the jack next to the still, directly under the glass arm (labelled 2 in the picture). Open the stopcock on the collection flask so that the toluene runs out of the collection bulb and into the bottle. When the collection bulb is empty, turn the stopcock back to the previous position, plug in and turn the power back on to continue distilling.
11. Make sure there is always enough undistilled solvent in the round bottom flask. **NEVER boil the flask dry.**
12. When you want to refill the round bottom flask with more toluene, unplug and turn off the power on the heating mantle and wait until the still has cooled. Fill as above (see point 7) and turn the heating mantle back on. **Never refill the flask while it is still hot.**
13. When you are finished distilling, turn the stopcock on the collection bulb so that any distilled toluene will run back down the column and into the round bottom flask and then **turn off and unplug** the heating mantle. The timer is only a preventative measure, you must unplug the still when you have finished. **Never leave the collection bulb full after use.**

14. **Quenching the still:** The flask should be completely cool and approx. 1/5 full. Add **isopropyl alcohol** (IPA) and leave overnight, then add **methanol** (gentle heating may be required to get all the calcium hydride to react, e.g. setting 2 on dial for 1 h). **After cooling, carefully add water** and clean the flask, column and still head. Quenching must be carried out during **normal working hours** in case a problem arises.

