**Purification of TMEDA:**

1. In a 4L Beaker, add, 2L MeOH and 500 ml TMEDA and stir.
2. Add, 550 ml of conc. HCl dropwise using a dropping funnel at 0 °C with stirring.
3. Once the addition is complete, allow the mixture to come to rt. Put the solution in the freezer overnight.
4. Filter the liquid and isolate the solid (TMEDA.2HCl).
5. Boil approx. 1.5 L of MeOH (Don’t boil vigorously) and add the solid with stirring.
6. Add 450-500 ml of water to dissolve the solid.
7. Allow the mixture to come to rt. Put the solution in the freezer overnight.
8. Repeat 4-7 again (This time, add 300-350 ml of water to dissolve the solid).
9. Filter the solid and allow it to dry overnight (open to air).
10. Add approximately 400 g of KOH (solid) in portion (200 g + 100 g + 100 g) to TMEDA.2HCl in 1L Erlenmeyer flask in an ice bath and stir it (Its hard to stir with a stir bar in the first place, so you might need to hand stir with a spatula occasionally it. But once the liquid starts forming, it becomes easier to stir with stir-bar.)
11. After 8 hr, remove the ice-bath and stir it overnight.
12. Decant the liquid on 10 g of KOH (solid) to further dry it and stir for 2 hrs.
13. Decant the liquid on CaH₂ and stir it for 2 hrs.
14. Decant the liquid to a 250 ml rb and add sodium metal to dry. Stir it overnight to dry.
15. Vacuum transfer the pure TMEDA to a 250 ml over-dried rb and add sodium and benzophenone to make the still.

[The final volume of pure TMEDA varies between 150 ml to 200 ml. This could be because of loss of TMEDA.2HCl during recrystallization step. Also, KOH DOES WORK BETTER THAN NaOH for this procedure.]

**How to make a still:**

**A good site to check:** [http://www2.chemistry.msu.edu/Safety/safety_sop_01.asp](http://www2.chemistry.msu.edu/Safety/safety_sop_01.asp)

1. Pour your solvent into a oven-dried 1L of round bottom flask. Add a stir bar to it.
2. Cut the one or two sodium cubes into small pieces. Add to the flask along with two spatulaful (approx. 10g/L of solvent) of recrystallized benzophenone (For hydrocarbon solvents, add 5-10 ml of tetraglyme/L of solvent to solubilize the ketyl radical).
3. Stir it under vacuum releasing the pressure time to time.
4. Once the solution turns blue, store it under vacuum.

[If after this, the solution does not turn blue, try adding more sodium/benzophenone or distilling the solvent to a different container and make a fresh still out of that.]