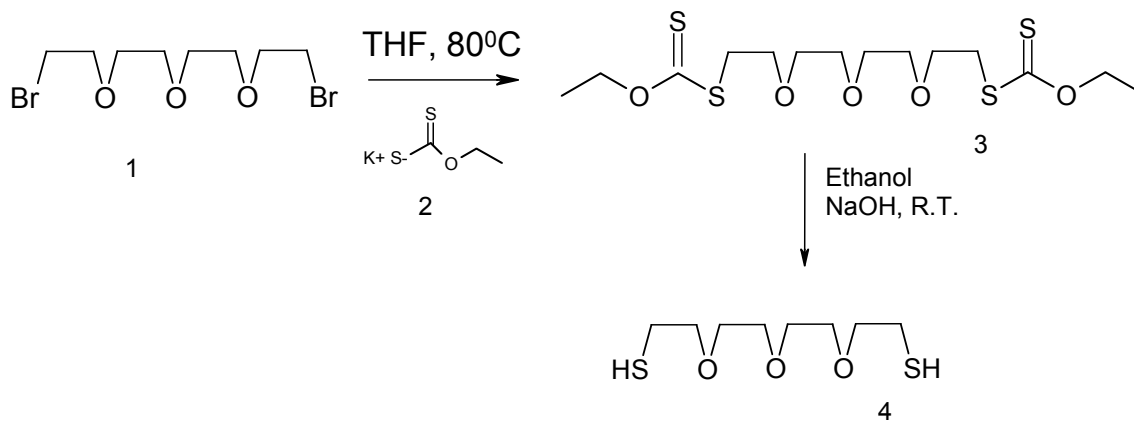


Standard Operation Procedure for Disposal of Unknown Thiols Venkat Donuru, (Dr. Liu lab)



Above reaction is a two step-one pot reaction to convert Bromine group to “SH” group. The reaction is expected to be air sensitive and to form unwanted disulphide bonds. The following procedures should avoid any unnecessary side reactions and at the same time destroy any water soluble thiols.

Reaction Procedure

First step reaction procedure :

Take 1 : 2.5 of Di Bromo tetra-Ethylene glycol and Ethyl Potassium xanthate in appropriate three neck round bottom flask containing THF with reflux set-up. Remove the air using vacuum and purge with nitrogen to remove traces of air. The reaction temperature is increased to 80°C. Monitor the reaction using thin layer chromatography technique (converts to non polar compound, visible under UV radiation).

After completion of first step, dilute the reaction mixture with dichloromethane and acidic water (water+5 drops of HCl). Separate the organic layer from aqueous layer. Wash the organic layer with water until clear organic layer appears. **Save all the aqueous layers** for further disposal treatment. Dry the organic layer with sodium sulphate and concentrate under vacuum to obtain **3**. To destroy the volatile thiols place NaOCl traps (Laundry Bleach~ 5% NaOCl or High-Test Hypochlorite (HTH) 30% NaOCl) in between the vacuum pump (self contained mechanical pump) and rotary evaporator as shown in Fig1. **Collect the pump water for disposal treatment.**

Second step reaction procedure :

Dissolve **3** in ethanol and remove air under vacuum. Add NaOH to the solution at 0°C and stir the reaction mixture at room temperature. The reaction is monitored using thin layer chromatography technique. Upon completion of the reaction, ethanol is removed under vacuum as described above and diluted with dichloromethane. The reaction mixture is washed with acidic water. The water layer may contain thiol impurities. **Save**

the water layer for disposal treatment. The organic layer is dried and concentrated and then used for further reactions. **Collect all the pump water for disposal treatment.**

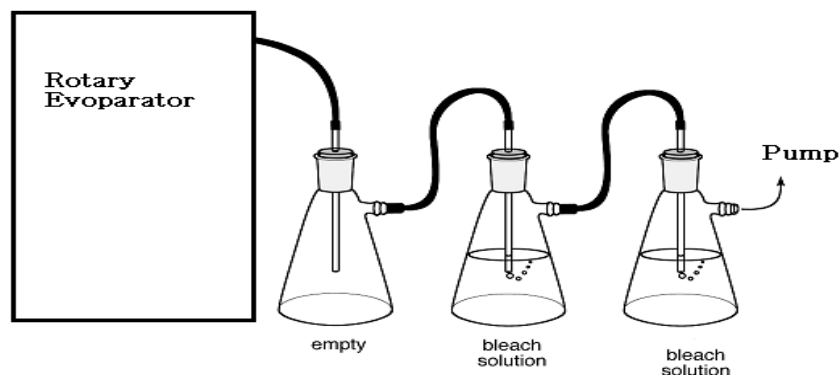


Fig 1: Schematic diagram of thiol traps for rotary evaporator

Disposal treatment of “thiols” in water:

All the “thiols impurities” in the above reaction are volatile, water soluble compounds which can be disposed of after treatment with sodium hypochlorite (Laundry Bleach or High-test hypochlorite). Thiols are converted into sulphonic acids upon treatment with sodium hypochlorite which can be disposed into drain after several fold dilution.



Procedure:

Collect all the wash layers into a suitable container and add slowly excess HTH solution in the hood (exothermic addition). Shake the container vigorously several times and then keep the container in the hood for 24 hours. After 24 hours test for thiol smell, if no such smell observed it can be disposed to the drain with excess of water.

References

1. www.books.nap.edu
2. www.chem.harvard.edu
3. Dr. Marshall W. Logue