Safe Working Procedure for Reactions in Sealed Tubes

A number of organic reactions are sometimes carried out in sealed tubes. This is typically to allow the use of a solvent above its normal boiling point. Before carrying out a reaction in a sealed tube, the use of a higher boiling solvent in a normal reflux apparatus should be considered. A specific risk assessment must be submitted for sealed tube reactions.

Hazard

Upon heating, which may be external heat or heat generated by an exotherm, or due to the generation of gases within the tube, the internal pressure will increase. There is the danger of an explosion. Information about a sealed tube explosion may be found at http://teamsites.ntu.edu.sg/ohs/Shared%20Documents/ALERTS/OHS%20Alerts/OHS%20ALERT%203%20Explosion%20of%20a%20chemical%20reaction%20vessel.pdf.

Precautions

All reactions in sealed tubes must be carried out in vessels made of a material that can withstand the internal pressure. The tube should be sealed using a suitably robust screw cap. A sealing device with a pressure release valve is strongly recommended. All apparatus must be carefully inspected for cracks and faults before use. Sealing glass tubes with a flame is not permitted without specific permission from the Division safety officer. The choice of vessel and sealing method must be reflected in the RA.

The reaction must be carried out in a well ventilated fume cupboard. In addition, the reaction should be behind or within an additional safety shield made of an appropriate material (e.g. polycarbonate, ≥7mm thickness).

At the end of the reaction, the excess pressure should be released gradually and with caution, after cooling to room temperature.

The following personal protective equipment (PPE) is to be used:

   Safety Glasses, Labcoat, Latex gloves, Long pants, Covered shoes

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