How to prepare liquid reagents without oxygen present

Many reactions will require that they be carried out in the absence of atmospheric oxygen and often moisture. It is for this reason that most of the research labs have stills to dry the commonly used solvents and collect them under a nitrogen or argon atmosphere.

To remove oxygen from a reagent by the same method can be somewhat tedious and wasteful. De-oxygenation can be achieved by one of the following three methods:

(i) Bubbling with an inert gas. Usually, sufficient oxygen can be removed from an involatile reagent by slowly bubbling a gas such as nitrogen or argon through the liquid. This is not a rigorous method, but is often applicable when larger volumes of reagent are to be used in a relatively insensitive reaction.

Simply put a disposable pipette into the end of a plastic or rubber tube attached to a nitrogen line. If you find it necessary to close any taps to the outlet bubbler, be sure to open them when you have finished.

(ii) Trap-to-trap distillation. This is ideal for small volumes of volatile material. It must be carried out on a vacuum line with two glass vessels that can be linked in tandem to the line. The vessel furthest from the vacuum line contains the reagent, frozen in liquid nitrogen and under vacuum. With the vacuum turned off (i.e. the glassware is isolated from the vacuum line) allow the reagent to warm to room temperature whilst the second vessel is cooled in liquid nitrogen. With a good vacuum and a fairly volatile reagent, appreciable amounts of liquid can be transferred efficiently. This method has the advantage that it can be adapted to (a) dry the reagent (put a drying agent in the first vessel); (b) keep the distilled product under a vacuum or gas of choice; (c) be used as a purification step for a dirty reagent. The method is limited by volume, availability of a good vacuum (the joints of the glassware must not leak!) and the volatility of the reagent (although a heat gun can often be a good persuader).

(iii) The freeze-thaw method. This is a universally useful method, but only as a method for removing oxygen (de-gassing). It cannot be used as a rigorous method for drying.

A vessel containing the reagent is connected to a source of high vacuum (a mechanical pump, not a water aspirator). With the tap to the pump closed, the reagent is frozen in liquid nitrogen by placing the vessel in a dewar so that the surface of the liquid nitrogen is level with that of the reagent. Whilst still sitting in the liquid nitrogen bath, the tap to the vacuum is opened and all volatiles are removed. The tap is then closed and the vessel is removed from the cold bath. After the reagent has melted, it is re-frozen and the tap is opened again. Repeating this cycle three times should be sufficient to remove the last vestiges of oxygen from the frozen matrix. After the final cycle, allow the vessel to warm to room temperature under vacuum *and then* slowly let in the inert gas of choice (nitrogen). Be careful to follow the suggested order of events as it is easy to condense solvent from another reaction on the same nitrogen line.