Reflux and distillation

Posted by Anonymous on Thu, 2015-04-30 11:46

Reflux and distillation: How should solutions be heated when using quickfit apparatus to perform reflux or distillation? Are Bunsen burners still allowed? We do not have any heating mantles.

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reflux and distillation

Submitted by sat on 13 May 2015

When performing reflux and distillation using Quickfit apparatus (standard taper ground-joint glassware) in a school, the safest and most practical method for heating reactants with boiling points below 100° C is the use of a water bath. When heating reactants with boiling points above 100° C, a heating mantle with a correctly fitting flask or a sand bath can be used. A Bunsen burner, or any other naked flame, is not advisable, unless heating non-flammable liquids.

Reflux and distillation:

Reflux and distillation are common techniques used in school chemistry classes. Quite often, these experiments require the heating of flammable organic solvents, whilst occasionally distillation is used to separate non-flammable inorganic solutions. The most common experiments are in carbon chemistry—refluxing of an organic reaction—esterification of carboxylic acids and the use of distillation to purify an ester and make boiling point (BP) determinations.

The flammable organic solvents that are used for these reactions include alcohols such as ethanol, methylated spirits, propan-1-ol and butan-1-ol. Most of the esters that are produced would fall into the highly flammable through to combustible classifications. All of these chemicals require careful handing especially in respect to exposure to heat sources. The major concern is the presence of flammable vapours in the vicinity of any naked flame. Vapours from organic solvents are heavier than air and can travel long distances along a bench or floor.

It is important to keep all naked flames and static discharge sources away from flammable organic solvents. Schools should ensure that they **DO NOT** use a Bunsen burner for heating a reaction when there are flammable organic solvents present, even with reflux or distillation setups. The Bunsen provides a very high heat source that could very quickly ignite organic solvents and vapours. It is also difficult to evenly distribute heat around any reaction flask with a Bunsen and there is a risk of cracking the reaction vessel and releasing the contents.

Recommended methods for heating flammable liquids:

Water Bath:

This is the recommended method for heating reactions up to 100° C using Quickfit apparatus during reflux or distillation procedures. For distillation, a water bath generally has to be 15° C higher than the boiling point of the liquid being distilled. It is important to check that the liquid you are distilling is suitable for this heat range. A water bath can be simply prepared by heating a beaker or pan of water using a hot plate. It is important to use a container large enough to hold the reaction flask and provide a buffer of water between the outside of the reaction flask and the beaker or container. This technique is recommended for the following reasons.

1. Absence of a naked flame.
2. Overheating is prevented.
3. Even and controllable heating is provided for both round bottom and pear-shaped flasks.
4. If the reaction flask were to crack, then the spilled contents would be captured and diluted in the water bath minimising the risk of ignition. It should be noted that the surface of the hotplate is a potential ignition source, if an organic solvent were spilt directly onto it and its flash point[1] exceeded. Never heat an organic solvent in an open container on a hotplate.
5. This technique is suitable for temperatures up to 100° C and is adequate for the majority of reactions conducted in school chemistry labs.

Steam bath:

A steam bath can be constructed using a vessel of water heated over a hotplate. Alternatively, electrically heated steam baths can be purchased through science equipment suppliers. These are fitted with constant water level devices and overlapping concentric rings, which can be removed according to the size of the vessel being heated. The ‘in-line’ style is useful for the classroom situation. Steam baths can be used to heat low-boiling liquids and are suitable for heating flammable liquids.

Heating Mantle:

This is the recommended method for heating solutions over 100° C using Quickfit apparatus during reflux or distillation procedures. Heating mantles are the best option for these temperatures as long as the flask is the **correct size to fit the mantle**. They are designed to be used with a properly fitted round-bottomed flask. A heating mantle that is not the right size will have poor contact with the reaction flask and the heating temperature will be very difficult to control. Care needs to be taken as the inside surface is a potential ignition source if an organic solvent were spilt directly onto it and the solvent’s flash point exceeded. A heating mantle is an expensive piece of laboratory equipment.

Sand Bath:

This is also a method that can be used for heating solutions over 100° C using Quickfit apparatus during reflux or distillation procedures. An iron or steel container is filled with clean, dry, washed sand and placed onto a hotplate. Do not use glass containers, as there is a risk of them cracking under the high heat. The reaction flask is placed into the sand which conforms to the shape of the flask. The sand conducts heat from the hotplate to the reaction vessel. The sand at the bottom of the bath will be hotter than the sand at the top. The flask can be raised or lowered in the sand bath to the desired temperature. Even heating is provided by this method. The temperature is monitored by a thermometer that is inserted into the sand. Sand baths can be heated to temperatures greater than 250° C. A disadvantage is that they take a long time to heat up and cool down. In addition, they can be very heavy and present a manual handling hazard. Avoid spilling any water into the bath, as this can result in hot sand being splattered out causing injury. There are bath surrounds that can be purchased that sit on top of the hotplates as a safety measure.

Other methods for heating flammable liquids:

There are other types of heating sources available in laboratories, such as oil baths and heat guns. However, these are less likely to be used in school science labs.

Oil Bath:

Oil baths are used for temperatures above 100° C. The bath is usually magnetically stirred and heated on a hotplate. Silicon oil and mineral oil are the two most common oils used. Silicon oils are non-flammable, do not give off unpleasant odours, and are very stable. Mineral oil is much less expensive, but is flammable. It should not be heated above 175° C.The silicon

fluids are probably the best liquids for oil baths, but they are very expensive for general use. In general, oil baths are difficult to handle. Electrically heated oil baths are available but are expensive. The following precautions need to be taken:

- The mineral oil should not be overheated, as it will smoke. If the flashpoint of the oil is reached, it can catch on fire.
- If heating over a hotplate, then you need to use a metal pan. A glass container may crack spilling the contents onto the hotplate.
- Oil baths should be mixed to prevent hot spots.
- Do not overfill as oil expands in volume when heated.
- Be careful not to tip over the hot oil, use a laboratory jack to support and raise or lower the bath.
- Do not allow water or any volatile into the hot oil, the water will boil and may splatter the hot oil out.

Heat Guns:

These get to very high temperatures very quickly (up to 500° C). They operate by sucking air across red-hot heating elements thereby increasing ignition risks in the presence of flammable organic solvents. The nozzle of a heat gun during operation is also an ignition source. Never use them near flammable liquids or environments containing flammable vapours. In addition, even heating is difficult to achieve by this method. They are generally used in the laboratory when a short burst of heat is required, and not as a heat source for reactions or distillations.

Use of Bunsen Burners:

A Bunsen burner should only be used to heat non-flammable liquids. Examples of distillations when Bunsen burners are safe to use include: steam distillations of essential oils, ink solutions, sea water and copper (II) sulfate solutions.

Safety Considerations:

Other safety issues that need to be considered with refluxing and distillation techniques to prevent accidents that could expose any flammable chemical to the heat source include the following.

- Check that any glassware used is free from chips or cracks.
- Keep Quickfit joints lubricated with a thin layer of vacuum grease and make sure they are firmly clamped with special joint clips/clamps, which are available through science equipment suppliers.
- Add boiling chips to the reaction liquid before heating to produce smooth boiling and prevent superheating.
- **Never heat a closed system** as the build up of gas may cause an explosion. Always check that there are no blockages between the mixture and the top of the condenser.
- Provide good ventilation and be aware that naked flames should not be lit until the reaction has totally cooled, equipment disassembled and removed from the lab and no vapours are present.
- Never use a flat-bottomed flask. A round-bottomed flask is better to use, as it is less

likely to crack.
- The reaction flask should not be more than half-filled with the reaction mixture.
- Use proper disposal methods for all wastes.

Science ASSIST recommends that a risk assessment be conducted and SDSs consulted for all chemicals used and produced. The risk assessment should also be conducted for all equipment used in the reflux and distillation procedures. Science ASSIST has developed a one-page risk assessment template that may be useful. See Risk Assessment Template.

Glossary:

Distillation:

Distillation is a technique used to purify and separate a liquid mixture. It is based on the different boiling temperatures of liquids. The liquid with the lower boiling point evaporates first (most volatile) and the vapours are then condensed back into liquid form in a sequence from lower to higher boiling points. The temperature is usually maintained at the boiling point of the lower boiling component of the mixture. Simple and fractional distillation is commonly used in school labs.

Reflux:

Reflux is a technique generally used to prevent the loss of any volatile reagent and solvent by evaporation. It is usually carried out at a constant elevated temperature. In school science laboratories, reflux is commonly used for the synthesis of esters where a mixture consisting of an alcohol, carboxylic acid and a small amount of concentrated sulphuric acid (catalyst) is heated in a vessel fitted with a water-cooled condenser to prevent loss of volatile material.

Quickfit glassware:

Specialised, interchangeable, borosilicate glassware, which have been designed with precision manufactured joints that fit together quickly and easily, providing good seals. Quickfit is a brand name and the glassware comes in many shapes and sizes. Quickfit glassware is commonly used for distillation and reflux techniques in chemistry laboratories.

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