

Safety Bulletin - Distillation of Methyl tert-Butyl Ether

1. Overview of Incident

Methyl *tert*-butyl ether (MTBE) was being distilled in an attempt to dry the solvent before use. During the distillation, solvent vapour was released from the apparatus (likely due to a build-up of pressure) and made contact with a heat-gun element being used to heat the still-pot. The solvent vapour ignited resulting in a fire. The researcher sustained a burn to the face and charring of the hair just above the forehead. Fortunately, the researcher was wearing a laboratory coat and safety glasses with hair tied back. After raising the alarm, the fire was extinguished with a fire blanket and CO₂ extinguisher.

2. Lessons Learned

During the accident investigation, several lessons were identified for future improvement.

2.1 Use of Safer Alternatives

Distillation from a drying agent is a well-established technique in synthetic chemistry, however, it may be possible to dry solvents to acceptable levels using activated molecular sieves. A paper by Williams and Lawton evaluates a variety of desiccants for drying common lab solvents (Drying of Organic Solvents: Quantitative Evaluation of the Efficiency of Several Desiccants; *J. Org. Chem.* **2010**, *75*, 8351). The approach removes the need to distil the solvent and eliminates the hazard at source. It is good practice to exhaust safer alternatives before carrying out a process with a greater intrinsic risk.

2.2 Hazards Associated with Heat Guns

Heat guns are often considered safe alternatives to Bunsen burners / microburners, however, this can create a false sense of security. Despite the lack of a naked flame, heat-gun elements can become redhot during use and act as an effective ignition source for flammable material. It is important to be aware of this hazard and treat heat guns with the same respect as a naked flame.

With regard to solvent distillation, heat guns should not be used as the primary heat source as controlling the rate of heating is more difficult than when using a hotplate / oil bath. Heating volatile solvents too vigorously may lead to a build-up of pressure within the distillation apparatus, leading to glass joints separating and the release of hot solvent vapour.

Accidents involving heat guns are not isolated and Princeton have reported similar incidents on their safety website (https://ehs.princeton.edu/node/382)

2.3 Removal of Flammable Materials

Related to point 2.2 above, when performing an experiment / operation that has a reasonable likelihood for an uncontrolled fire to develop (e.g. distillation, work with pyrophoric reagents), flammable material should be removed from the immediate vicinity. If a fire were to develop in close proximity to (i) waste solvent containers, (ii) flasks containing solvents or (iii) paper towels they could ignite, intensify the fire and allow it to spread rapidly.

2.4 Operational Scale

Scale is often a major contributor to the overall risk posed by a given process. If you are scaling a process, give consideration to the safety implications of this increased scale and introduce additional controls if required. In the case of distillations, it is important to consider whether the condenser is capable of providing sufficient cooling to handle the amount of solvent vapour being generated. Insufficient cooling may lead to pressure building within the apparatus and accidental release of solvent vapour.

2.5 Fire Safety Awareness

It is strongly encouraged that all researchers make themselves aware of the locations of fire extinguishers / fire blankets within their laboratory as this will help ensure response is as rapid as possible. Researchers should also make themselves aware of the nearest fire call point so they can raise the alarm in an emergency.

2.6 Risk Assessments

Risk assessments for general laboratory operations (e.g. distillations) can be easily overlooked, however, it is important that the risks associated with these operations have been considered and a risk assessment is in place before work is carried out.