Technical Guidelines for Safety in Chemical Laboratory Courses

Introduction

An important goal of chemical education of students of science disciplines is learning the risks and gaining experiences while working with hazardous substances in chemical laboratory courses. Students shall be introduced to make their own decisions for safety measures at each level of their education. The risk awareness resulting from this approach is another goal of these laboratory courses and has the same importance as learning complex scientific contexts and gaining knowledge on most recent technical developments.

The aim of a modern education must be, therefore, to learn safe handling of chemicals in laboratory courses as well as the circumspection to protect oneself and other persons working in the laboratory from risks. Furthermore, it is necessary to behave responsible to the environment and the public by following related guidelines and instructions. In order to avoid the formation of laboratory wastes it is useful to reduce the batch volumes. Multi-stage syntheses can be applied and the products can be used as starting materials for other experiments in order to save chemical resources.

Many laws, directives, regulations, and guidelines exist to rule almost all fields of safe handling of hazardous substances and equipments. But it is also necessary that the safety awareness imparted is based on fundamentals of chemistry in order to avoid accidents in laboratories. Annual statistics of accidents show that accidents are caused only in few cases by defects of technical installations and equipment. In most cases (about 80 %) result from human failures, whereby the lack of knowledge of the risks of hazardous substances and missing understanding of the reactions conducted play an important role. Accidents often happen because of the habitual behaviour of the operators resulted from long-term application of potentially dangerous methods and, therefore, neglecting safety measures needed.

To reach the goals described above it is essential to formulate a specific operating instruction for any laboratory course. They can be based on general instructions for laboratories. The operating instructions shall contain the specific aspects of the particular lab courses and additionally provide following information:

- A list of all chemicals used in the laboratory course including their R & S phrases (except substances to be analyzed in analytical courses, since that would contradict the teaching goals)
- Operating manuals for all apparatuses if they are not introduced in verbal instructions,
- detailed experimental and operating instructions or literature citations.

This operating instruction for the laboratory course must be read by all students and signed as taken note.

Beginners of chemistry, pharmacy, and biology shall be introduced into safe working before starting their chemical laboratory courses or conducting particularly critical experiments. This can be done either in an introductory pre-course or even at the beginning of the laboratory course itself. In the sequel of their studies the students shall be introduced at the beginning of each course if hazardous substances are involved. Previous knowledge can be expected as being familiar with. Beginners should gain the necessary informations of an operating instruction through the responsible teacher.

For safety measures it is necessary to know which emergency equipment is provided and where they are located in the laboratory. Important items are:

- Emergency exits, fire escape, and escape routes in general
- Alarm systems, telephone, and further emergency call equipment
- Extinguisher, fire alarm, and fire blanket
- Respiratory mask and related filters, safety shower, and eye wash
- First aid kits, stretcher, first aid room, and offices of the lecturer (to be called)

It is questionable whether all students and staff in the laboratory really know in case of emergency

- which of the chemicals handled with are explosive, toxic, or highly flammable,
- who does turn off the gas, water, power, and other feed lines and in which way
- elevators and fume hoods shall not be operated in case of fire,

- the extinguisher shall be refilled after each use,
- pressurized gas cylinders shall be safeguarded against falling down,
- what does the term “self-protection” mean,
- which emergency measures have to be taken in case of a serious accident,
- where safety informations can be found if needed

Before starting dangerous experiments students working quite near should be introduced into particular risks and safety measures, as well. This should be particularly considered in chemical laboratories if several students are working at the same fume hood.

General Measures for personal health protection

Some principle items have to be observed in order to ensure personal health protection:

- While working in the laboratory suitable work clothes should be worn. For regular laboratory experiments it is sufficient to wear a long-sleeved lab coat made of a non-melting material (preferentially cotton or a blended fabric of polyester and cotton). The lab coat should not be worn in other rooms than in the lab, e.g. in lecture rooms, libraries, catering rooms, etc., in order to avoid contamination with adherent chemicals.
- Stable and closed shoes should be worn.
- During all the time in the laboratory safety glasses with side-protectors should be worn.
- While performing an experiment student should not leave the laboratory if a continuous monitoring is needed and no other experienced person is present who does know the course of the experiment and is able to supervise it. In case of dangerous experiments at least two persons should be present.
- In the area of laboratories no foods or beverages should be stored nor consumed due to risk of contamination.
- Due to the risk of mix-up vessels which are usually designated for foods or beverages shall not be used to store chemicals, and vice versa (foods or beverages should not be filled in vessels usually used for chemicals).

- Smoking is not allowed in laboratories due to the risk of inhalation of contaminated smoke like it is the case for food, and due to the risk of ignition of fire and explosion with flammable chemicals.

Protective measures while handling dangerous substances and formulations

In principle, while handling dangerous substances and formulations any contamination or endangering of men and the environment as well as arise of dangerous situation must be excluded. In order to ensure this it is necessary that all persons handling with chemicals have sufficient knowledge on the following items:

- How are chemicals stored appropriately?
- Which vessels are suitable for safe storage of chemicals?
- Under which conditions chemicals shall be stored in fume hoods and which safety measures have to be taken?
- How can the (often dangerous) mix-up of chemicals be avoided?
- How can chemicals be transported without risk of breakage of the vessels and release of the chemicals?
- How can the spillage of chemicals or contact with the skin during taking them out of vessels be avoided?
- Which measures have to be taken if chemicals are spilled of or released in another way?

Far-reaching regulations for handling hazardous substances are set in force in Chemicals Acts (e.g. German ChemG). These laws shall protect men and the environment against the hazardous effects of chemical substances and formulations, especially to identify these effects, to avert them, and to avoid their occurrence. The Chemical Acts shall ensure that new substances shall be tested for their hazardous properties before being introduced into the market. According to the test results necessary safety measures shall be taken during production and handling of these substances. The results of these tests are the basis for the labelling of hazardous substances, i.e. for the assignment of danger symbols, giving hints to special risks, and deriving safety recommendations.

Storage of chemicals

Chemicals should be stored in the original packing of the supplier, if possible, since the etiquettes give valuable informations due to the danger symbols and R&S phrases. If other vessels are used they have to be labelled in the same way. In order to protect the etiquettes against the influence of the chemicals and to keep them legible they should covered with a transparent plastic sheet. The inscription should be light-resistant and written with a pencil or a permanent ink.

Vessels and jars for the storage of chemicals should be made of suitable material. Plastic or glass vessels are often used for this purpose. For the storage of light sensitive substances like diethyl ether which tends to form dangerous peroxides dark glass bottles should be used. If plastic bottles are used it should be taken into account that they may embrittle under the influence of sunlight and thus may break down. These bottles should be regularly checked and the chemicals transferred to another vessel, if necessary. Particular attention has to be given to the possible migration of organic solvents through plastic bottle walls.

Unnecessary stockpiling of chemicals above regularly needed amounts should be avoided. All substances stored in the laboratory should be checked at regular intervals, at least once a year. Chemicals which may release toxic, corrosive, or flammable gases or dusts shall be stockpiled only in small amounts in a fume hood.

Transportation and transfer of chemicals into other vessels

During the transportation of chemicals attention should be paid to avoid a break down of the vessels and release of the substances. Large full glass vessels are particularly subject to break down. They never should be carried at their neck, but transported in a bucket, rack, mobile trough or basket.

Transfer of chemicals from one into another vessel always implies the risk of spillage and, therefore, contact with the skin and contamination of clothes. Additionally, gases and dusts can be inhaled. The latter can also catch fire if no measures are taken against electrostatic charging.

In order to reduce these risks suitable funnels for liquids or powders should always be used, even if the operator is sufficient skillful to handle chemicals without auxiliary devices. During filling the liquids, but particularly toxic or corrosive ones, receiving tanks like troughs should be used regularly. The same has to be done while filling solids and powders, a receiving pad e.g. paper sheet should be used.

Under no circumstances, it is not allowed to pipette liquids by suction with mouth, since many accidents happened by doing this including poisoning and cauterization. This attitude should also be followed in case of harmless liquids in order to avoid a wrong habit in daily laboratory work. For filling liquids by pipettes suitable aids like suction balls should be used.

Important aspects for safe conducting of experiments

In order to reduce risks laboratory experiments must be planned and conducted carefully. For this purposes operating instructions are useful. They should not only contain the specifications of the reaction but also the labelling of the substances applied. Further important informations needed are possible risks for men and the environment, the protection measures and instructions about first aid in case of emergency, and informations on possible disposal measures for wastes.

Before beginning an experiment it should be checked whether there is enough time to conduct the entire experiment. Otherwise it must be decided if the experiment can be interrupted safely at certain time and without significant disadvantages.

All chemicals and equipment needed for a safe performance should be provided from very beginning. It should be strived for working with chemicals in a fume hood. Experiments must be performed in a fume hood if toxic or corrosive substances are applied and/or gases, vapours, or aerosols might be released in dangerous concentrations. An example for this is evaporation or fuming off substances as well as heating in open oil bathes.

To ensure a good performance of the fume hood the front gate and the side windows are kept closed during the course of the experiment. The full power is only possible if the air flow is not disturbed. This can best be reached by removing all flasks and jars from the working place in the hood if they are not needed. Flow disturbances can also arise from heat sources.

Particularly open flames like Bunsen burner have a significant influence on the performance of hoods and should, therefore, be avoided.

Since chemicals are usually hazardous they should not get in contact with the skin while handling. Dangerous substances should be handled only in small portions, and suitable protective gloves should be worn.

In student's lab courses no carcinogenic, mutagenic or teratogenic substances shall be applied. These substances shall principally be substituted by less hazardous ones if the teaching goals can be reached from the didactical, methodological, and scientific viewpoints. Exceptions from the "duty for replacement" can only be made if the particular experiments are of great significance for the daily practice of the subject. In fundamental lab courses for undergraduates those experiments should only be conducted at the end when the students developed sufficient experimental skills and are particularly introduced..

During heating of liquids special attention should be given to uncontrolled boiling up and squirt out of the jar by using boiling stones or magnetic stirrer. While heating liquids in test tubes they should be permanently shaken in order to avoid delay in boiling which otherwise could eject the whole content of the tube. For safety reasons the opening of the tube should never be directed to oneself or to other people nearby. If necessary squirted chemicals must be immediately disposed of, e.g. by neutralization of acids or bases first and then wiping the liquid using protective gloves.

Safety aspects of using equipment and apparatus during the experiments

Most chemical experiments are performed in glass apparatuses. Glass has many advantages in chemical experiments. It is not only non-reactive but it allows also the visual observation of the reaction course. But it is easy breakable and can thus cause accidents. Cuts and gashes from broken glass apparatuses are one of the most occurring injuries in laboratories. Breaking of equipment erected with glass components can release dangerous substances and may cause fire. The erection of glass apparatus must, therefore, be done under observing safety instructions. The use of unsuitable parts have to be avoided, e.g. different glass types, non-fitting ground-glass joints, etc. Complex glass apparatuses should be erected without

mechanical stress which might lead to break down. They should be placed on a safe place (best in a fume hood) and secured against upset.

Laboratory apparatuses are usually erected as an open system to the atmosphere in order to ensure a pressure compensation and to avoid explosion, except autoclave reactors made of steel or non-corrosive metals.

In many cases electrical devices are used as auxiliary materials, like stirrer, heater, centrifuge, etc. These equipments shall be in good technical conditions and meet the safety specifications for working with electricity. They must be checked in regular intervals by an expert technician in order to repair broken cables, plugs, contacts, etc., or to put out of operation if destroyed. This safety inspection is necessary for mobile as well as for stationary equipment. Drives and stirrer are usually operated with electric motors. They are usually not explosion-proof. In experiments applying highly flammable substances like hydrogen gas or hydrogen sulphide the electric motors are replaced by water turbines or air motors

Before starting an experiment all auxiliary parts at an apparatus have to be tested for working. This include, e.g., vacuum pumps, cooling system, stirrer, and other electrical drives – **before feeding the apparatus with the chemicals!**

Heating and cooling

As heat sources Bunsen burner, electrical heating plates, heating mantle, and heat bathes can be used. In case of highly flammable substances open flames shall not be used. The use of heating bathes is a safe method of heat transfer. They allow a heat transfer at low differences of the temperature. By using a heat bath the vessels should only be filled up to a certain height, since the heat transfer liquids are subject partly to a significant thermal expansion when heated. Furthermore, the heat transfer liquids and the substances to be heated shall not react with each other in a dangerous way if the reaction apparatus breaks down during the experiment. This does mean in practice that, e.g., suspended sodium or potassium metals shall not be heated in a water bath.

As a principle rule the heat source should be placed in such a way that it could be removed easily and without any alteration at the reaction apparatus. A suitable method for this is the use of a scissors lift plate.

While heating apparatus containing flammable substances condenser must be used. If these condensers are operated with water the hose couplings both for feeding and draining must be safeguarded with hose clamps. It must be taken care that cooling is maintained without interruption during the course of the experiment in order to avoid a dangerous fire accident or even an explosion. In experiments where alkaline or alkaline-earth metals or metal hydrides are applied the glass condensers should be substituted by the more stable metal condensers.

As coolants in laboratories ice, freezing mixtures of ice with salts (NaCl -21 °C, calcium chloride -55 °C), freezing mixtures of dry ice with solvents (-78 °C), or liquid nitrogen are regularly applied. They are handled in Dewar vessels for thermal insulation. Dewar vessels are thin-walled evacuated (high vacuum) hollow glasswares which can easily implode. Their top edges are particularly endangered. Therefore, Dewar vessels should be covered with a jacket of metal, tear strength plastic, etc. and the operator should wear protective gloves.

Flammable liquids should only be stored in refrigerators or freezers if they are constructed as explosion-proof.

Working under reduced pressure and in vacuum

While working in a laboratory reduced pressure or vacuum are often applied, e.g., for the distillation of easily decomposing substances or drying chemicals in desiccators. During the evacuation a pressure of about 1 kg/cm^2 rest on the glass surface due to the atmospheric pressure. This pressure stress can lead to an implosion if the glass apparatus is unsuitable for vacuum application or it is cracked at the surface (even almost invisible cracks). By implosion glass fragments are thrown around and may injure heavily the persons nearby (eyes, arteries)! It is, therefore, urgently needed, that an effective chip guard is used against implosion, e.g., as protective shield, desiccator cage, etc., particularly in case of large volume vacuum apparatuses

Flat bottom glass vessels like an Erlenmeyer flask shall never be evacuated due to acute risk of implosion!

It should be noted that vacuum formed with a water jet pump or a diaphragm pump is not less dangerous than that of a high vacuum pump. The pressure stress on the glass surface is almost the same in both cases. Even the relatively moderate vacuum applied in sucking (Büchner) flasks for filtering precipitates produces a pressure of 300-800 g/cm^2 on the glass surface.

A prompt aeration of heated evacuated apparatuses should be avoided, since the vapour-air mixtures produced inside may lead to an explosion.

Working at increased pressure

Reactions at increased pressure (overpressure) shall only be conducted in suitable pressure-proof vessels. These pressure vessels (e.g., bomb tubes, autoclaves) must meet the regulations of the "Pressure Vessel's Ordinance" (Druckbehaelterverordnung) regarding construction, erection, and operation, partly in special high pressure laboratories. Sealed tubes should not be removed out of the metal mantle nor the oven while pressurized.
Operation of autoclaves are performed in special laboratories and they must be inspected regularly in order to ensure their operations safety. Pressure and temperature limits given by the producers shall never be exceeded.

Drying of laboratory devices

Drying ovens in chemical laboratories are usually not designed explosion-proof and are not connected to the exhaust air systems. Laboratory devices should only be dried in these ovens after being thoroughly cleaned and then rinsed with water.

For drying chemical goods and products which may release flammable gases or vapours and, therefore, form dangerous or explosive mixtures only explosion-proof drying ovens shall be used.

References (only German)

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