

Laboratory  
Techniques in  
**Organic  
Chemistry**

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**I.K. International**

**LABORATORY TECHNIQUES IN  
ORGANIC CHEMISTRY**

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## Preface

This book deals with general information about work in Organic Chemistry Laboratory, viz., safety, first aid, different types of apparatus and their assemblies used for various types of reaction, stirring arrangements, heating techniques and low temperature experiments.

Various methods used for purification of organic compounds have been described. Besides the normal technique, the book includes write-up about molecular distillation, chromatography and electrophoresis.

Special emphasis has been given to the methods which can be used for working up of organic reactions. Various methods which can be used successfully for isolation of products from natural sources have been incorporated. Emphasis has also been given on the isolation of products from oily mixture using the technique of Liquid-Liquid extraction.

Methods for determining the criteria of purity of organic compounds have been discussed. The book also deals with drying and purification of solvents, preparation of spectroscopical grade solvents and HPLC solvents. The preparation of commonly used deuterated solvents (which are used for NMR spectroscopy work) is a special feature of this book.

**V.K. Ahluwalia**  
**Pooja Bhagat**  
**Renu Aggarwal**

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## General Information

This chapter provides general information for carrying out experiments in the organic laboratory. It describes the possible accidents in the laboratory and precautions to avoid them, the apparatus used in the laboratory for various reactions, viz. heating, cooling and stirring-techniques used in the laboratory, generation of gases and removal of solvents etc.

### 1.1 SAFETY IN THE LABORATORY

Chemistry laboratories are associated with several potential hazards and therefore certain precautions must be taken to minimize the probability of an accident. The knowledge of these hazards is the most important factor to avoid accidents and hence ensure safety in the laboratory.

#### 1.1.1 General Safety Considerations

The following are the general factors, which must be adopted by an individual to avoid accidents:

- (1) A thoughtless, careless and hurried behaviour must be avoided otherwise it may lead to an accident. Rather a thoughtful, careful, methodical approach must be adopted by an individual.
- (2) The laboratory must always be kept neat and clean. Anything drops at bench or floor should be cleaned up immediately so as to avoid slipping.
- (3) Running, eating, drinking or smoking in a laboratory should be avoided.
- (4) Safety glasses (goggles) and laboratory coat must be worn at all time in the laboratory.
- (5) Never work alone in the laboratory. The overnight experiments must be carried out in a special overnight room with necessary instructions.

## 2 Laboratory Techniques in Organic Chemistry

- (6) The position of first-aid box, exits and fire-extinguishers must be known by the person working in the laboratory.
- (7) Chemicals should always be kept in their appropriate place in the shelves.
- (8) Label should be read carefully before use. If the bottle is not labelled then either the content should be identified and then relabelled or it should be disposed of safely.
- (9) Cracked or broken glass apparatus should not be used.
- (10) Glass apparatus should also be stored at a proper place and not allowed to heap on the benches.
- (11) The waste material should be disposed of regularly.
- (12) Since most of the laboratory chemicals are poisonous, they should not be tasted and suction bulb should be used to suck them in filling pipettes.
- (13) To observe the odour of the component, it should be kept at a safe distance from the face and gentle fan of the vapour should be done with the hand towards the nose.
- (14) During heating, the mouth of the container should be away from the person and others nearby.
- (15) Hot objects should not be picked up by hands. Tongs must be used.
- (16) Before leaving the laboratory, the gas line should be closed properly.
- (17) All the accidents must be reported to the Instructor immediately.

### 1.1.2 Fire

The most obvious hazard in the organic chemistry laboratory is the fire, as most of the liquids used, particularly solvents, are quite volatile and inflammable. The most common reasons for a fire are :

- (a) Boiling of an inflammable solvent with a flame (without using condenser).
- (b) Using the inflammable solvent near a burner.
- (c) Using cracked apparatus, for inflammable liquids or cracking of the apparatus (containing inflammable liquids) while heating due to the faulty apparatus or unsuitable method of heating, and
- (d) Adding pumice stones to a heated liquid, which is bumping violently, so the liquid comes out and catches fire. Therefore,

always add pumice stones after cooling the bumping liquid for a few minutes.

When fire is large, go away from the burning area and bring it to the notice of the instructor immediately. The spread of fire can be prevented by removing the containers of inflammable solvents from the area and turning off any burner, if possible.

#### **1.1.2.1 Burning clothing**

If clothes catch fire, do not allow to run the victim as rapid movements fan the flames. The person should be made to lie down on the floor and fire proof blanket is wrapped around the burning clothes until the fire is extinguished.

In laboratories, sufficient number of fireproof blankets should be available. Each blanket should be kept in a labelled box (without lock) so that it can be taken out from the box without any delay. Alternatively the person whose clothes are set ablaze, should stand below a shower (which should be provided in the laboratory).

#### **1.1.2.2 Bench fires**

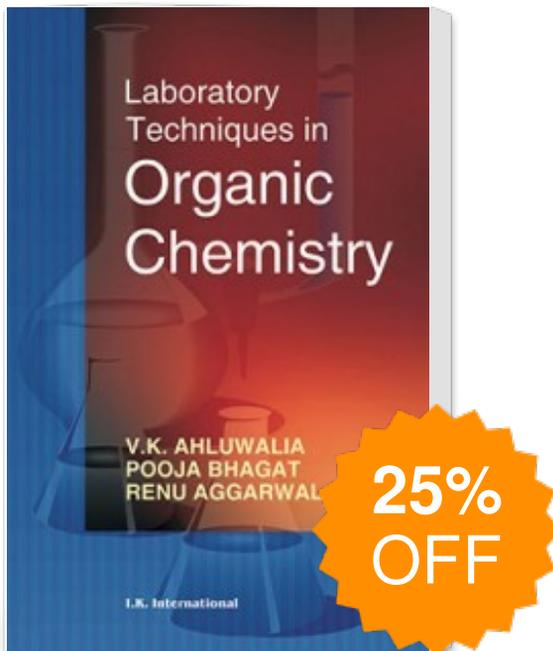
All the gas burners and the electrical hot plates near the area should be turned off and the inflammable material, if any, should be removed immediately.

Following methods are used to extinguish bench fires:

- (A) A small fire can be caused by burning of a liquid in a flask, beaker or in an oil-bath. It can be extinguished by stopping the flow of air in the container, which can be done by covering the opening of it with a clean wet cloth or duster. And the compound or the reaction mixture can be recovered.
- (B) **Dry Sand:** Most of the fires in the laboratory can be suppressed by the abundant use of dry sand. Laboratories should be equipped with buckets of dry sand properly distributed throughout and especially reserved for this purpose. After use, the sand should be thrown away and not reused as it is now contaminated with the inflammable, non-volatile materials.

So, for extinguishing fires, the sand is of great importance but it has some drawbacks also:

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