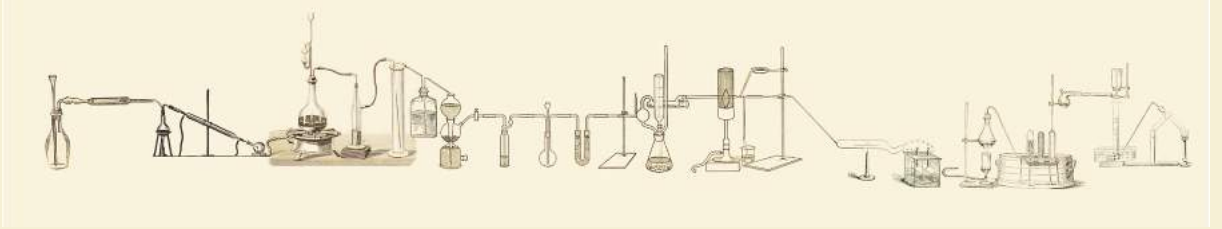
Laboratory Exercises in Identification of Organic Compounds

THE JOHN PAUL II CATHOLIC UNIVERSITY OF LUBLIN
FACULTY OF NATURAL SCIENCES AND HEALTH

WYDAWNICTWO
KUL

Oleg M. Demchuk, Małgorzata Topyła,
Katarzyna Kapłon, Karolina Mikulska



Laboratory Exercises in Identification of Organic Compounds

Wydawnictwo KUL, Lublin 2021

Reviewers
dr hab. inż. Radomir Jasiński, prof. PK
dr hab. n. med. Maciej Mastyk, prof. KUL

Typesetting
Jarosław Łukasik

Cover design
Agnieszka Gawryszuk

ISBN 978-83-8061-993-7

Wydawnictwo KUL
ul. Konstantynów 1H, 20-708 Lublin, tel. 81 740 93 40,
e-mail: wydawnictwo@kul.lublin.pl, http://wydawnictwo.kul.lublin.pl

Druk i oprawa
volumina.pl Daniel Krzanowski
ul. Księcia Witolda 7-9, 71-063 Szczecin, tel. 91 812 09 08, e-mail: druk@volumina.pl

TABLE OF CONTENTS

INTRODUCTORY WORD

The "classical" chemical analysis has been a valuable source of information on organic compounds' structure as well as their chemical reactivity for generations. It is worth mentioning that recently, in the workshop of organic chemists, using advanced spectral techniques to solve simple analytical problems has become increasingly dominant. This methodology vividly resembles "using lasers to kill a fly." It is often neither the simplest nor the cheapest or the most effective one.

Regrettably, "classical" methods of analysis of organic compounds are discussed less and less in academic textbooks. This publication addresses the existing gap. It contains a very precious study which may fascinate not only students but also young university employees. It will certainly help to conduct classes in chemical analysis effectively. It can also be helpful in the implementation of diploma theses and research work in various research units and institutions.

Radomir Jasiński

ACKNOWLEDGEMENT

This book was prepared in collaboration with the Scientific Circle of Biotechnology Students of the The John Paul II Catholic University of Lublin.

Authors are grateful to the Student Council and the University Authorities for the financial support with the USS grant.

INTRODUCTION

The identification of organic compounds belongs to the primary activities not only ofof organic chemists but also people not related to chemistry in their everyday lives. For instance, a commonplace distinction between table salt and sugar or sodium hydrogen carbonate is a simple organoleptic identification process. The same situation is when we check if our dishes are adequately cooked or seasoned - this is a process of subconscious identification of the presence or absence of particular organic compounds in the tested "reaction mixture."

Industrial development has placed more demanding challenges ahead of us, such as identifying substances hazardous to the environment or undesirable compounds (impurities) in drugs. However, chemists, mainly organics, will be interested in the methods of analysing the products of chemical reactions.

The primary purpose of writing this textbook "Laboratory exercises in identification of organic compounds" is a practical help for students to develop the ability to independently identify simple organic compounds based on qualitative analysis data, elemental analysis, and available spectroscopic data, including ^{1}H, ^{13}C NMR and mass spectroscopy.

It is necessary, first of all, to recall basic compound purification techniques such as crystallization, distillation (including reduced pressure), steam distillation, extraction, and sublimation, assuming that unidentified organic compounds may have impurities that prevent proper identification of functional groups and performance of the reliable quantitative analysis. These procedures are already well covered in the introductory organic chemistry laboratory course and therefore should not take more than two introductory classes to recall. We recommend thus, that you carry out the separation of one of the simple binary mixtures of liquid compounds: a) $PhNH_2$ / $PhNO_2$, b) Et_2O / EtOAc, c) $PhNO_2$ / $CH_3(CO)Ph$; purification of solids by crystallization and sublimation of a) 1-Br,4-NO_2-C_6H_4, b) cinnamic acid, c) aspirin, d) benzoic acid.

After a quick reminder of the primary methods of purification of organic compounds, one should start the independent analysis of the "unknown" substance received from the laboratory teacher (about 15 g) (the sample may contain about 10% impurities) or a mixture of substances.

After preliminary sample purification, several qualitative tests must be carried out to identify the functional groups and heteroatoms present in the compound. The solubility group of a compound, melting or boiling point, refractive index, and density should also be determined. The results obtained should be kept up to date in the final report (specimen at the end of the book) and the laboratory journal.

If necessary, it is recommended to use additional materials, which should be available from the teacher. These are: CHN analysis data, NMR ^{13}C, ^{1}H; IR, UV, and MS spectra, PROVIDED THAT A DETAILED SPECTRA ANALYSIS IS INCLUDED IN THE FINAL REPORT.

The analysis of the obtained results should allow for unambiguous identification of the substance obtained.

During the class, the tutor provides the necessary instructions on the techniques of performing individual experiments and will supervise the observance of health and safety rules.