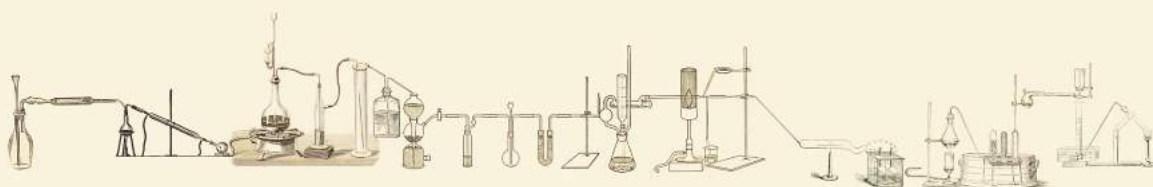


Laboratory Exercises  
in Identification  
of Organic Compounds

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



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# Laboratory Exercises in Identification of Organic Compounds

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# TABLE OF CONTENTS

INTRODUCTORY WORD .....	9
ACKNOWLEDGEMENT .....	10
INTRODUCTION.....	11
SAFETY ISSUES.....	13
<b>1 DETERMINATION OF MASS AND MOLAR REFRACTION .....</b>	<b>14</b>
<b>2 DENSITY DETERMINATION .....</b>	<b>17</b>
<b>3 IDENTIFYING OF A SOLUBILITY GROUP .....</b>	<b>19</b>
<b>4 COMBUSTION TEST .....</b>	<b>23</b>
<b>5 FUSION WITH SODIUM TESTS.....</b> <i>(test for heteroatoms)</i>	<b>25</b>
<b>6 TURIN DETERMINATION OF ORGANIC CARBON CONTENTS.....</b> <i>(organic compounds)</i>	<b>29</b>
<b>7 DISTINCTION BETWEEN SATURATED AND UNSATURATED ALIPHATIC HYDROCARBONS .....</b> <i>(hydrocarbons)</i>	<b>31</b>
<b>8 REACTION WITH BROMINE WATER .....</b> <i>(alkenes, alkynes, phenols, anilines)</i>	<b>33</b>
<b>9 BAEYER TEST .....</b> <i>(alkenes, alkynes, phenols)</i>	<b>34</b>
<b>10 REACTION WITH SODIUM.....</b> <i>(active hydrogen detection)</i>	<b>36</b>
<b>11 TEST WITH SOLUTION OF IODINE.....</b> <i>(unsaturated compounds, compounds containing oxygen atoms)</i>	<b>37</b>
<b>12 ALUMINIUM CHLORIDE IN CHLOROFORM TEST .....</b> <i>(aromatic compounds)</i>	<b>38</b>
<b>13 FUMING SULPHURIC ACID TEST.....</b> <i>(aromatic compounds)</i>	<b>39</b>

<b>14 TEST WITH SILVER NITRATE SOLUTION</b> .....	40
<i>(alkyl halides, acyl halides, carboxylic acids, sulphonic acids, chlorides of sulphonic acids)</i>	
<b>15 TEST WITH SODIUM IODIDE SOLUTION</b> .....	41
<i>(alkyl, sulphonyl, acyl, allyl, and benzyl halides)</i>	
<b>16 ACETYL CHLORIDE TEST</b> .....	42
<i>(alcohols, amines)</i>	
<b>17 JONES REAGENT TEST</b> .....	43
<i>(alcohols, aldehydes, enols)</i>	
<b>18 LUCAS TEST</b> .....	45
<i>(difference of 1°, 2° and 3° alcohols)</i>	
<b>19 CONCENTRATED HYDROCHLORIC ACID TEST</b> .....	47
<i>(tertiary alcohols)</i>	
<b>20 IODOFORM TEST</b> .....	48
<i>(aldehydes, ketones, alcohols having a <math>CH_3</math> group in an <math>\alpha</math>- position in relation to the carbonyl group)</i>	
<b>21 TEST WITH IRON (III) CHLORIDE SOLUTION</b> .....	49
<i>(phenols and enols)</i>	
<b>22 LIEBERMANN TEST</b> .....	50
<i>(phenol)</i>	
<b>23 PHOSPHORUS(V) CHLORIDE TEST</b> .....	51
<i>(alcohols, phenols and carboxylic acids)</i>	
<b>24 ESTRIFICATION OF ACETIC ACID</b> .....	52
<i>(alcohols)</i>	
<b>25 TEST FOR THE DETECTION OF POLYOLS</b> .....	53
<b>26 BORAX TEST</b> .....	54
<i>(carbohydrates, 1,2-diols, polyols)</i>	
<b>27 FERROX TEST</b> .....	55
<i>(oxygen containing compounds)</i>	
<b>28 TEST WITH 2,4-DINITROPHENYLHYDRAZINE</b> .....	56
<i>(aldehydes and ketones)</i>	
<b>29 TEST WITH TOLLENS REAGENT</b> .....	57
<i>(aldehydes, carbohydrates)</i>	

<b>30 FEHLING REAGENT TEST</b> .....	58
<i>(aldehydes, carbohydrates)</i>	
<b>31 SODIUM BISULPHITE TEST</b> .....	59
<i>(aldehydes and ketones)</i>	
<b>32 TEST WITH SOLUTION OF HYDROXYLAMINE</b> .....	60
<i>(aldehydes and ketones)</i>	
<b>33 BENEDICT REAGENT TEST</b> .....	61
<i>(aliphatic aldehydes, carbohydrates)</i>	
<b>34 TEST WITH PHENYLHYDRAZINE</b> .....	62
<i>(carbohydrates, vicinal di- and polyols)</i>	
<b>35 OXIDATION TEST WITH ORTHOPERIODIC ACID</b> .....	64
<i>(carbohydrates, 1,2-diols, compounds <math>\alpha</math>-hydroxycarbonylic and <math>\alpha</math>-hydroxycarboxylic)</i>	
<b>36 THE MOLISCH TEST</b> .....	66
<i>(carbohydrates and glycosides)</i>	
<b>37 THE SELIWANOFF TEST</b> .....	67
<i>(distinguishing aldoses from ketoses)</i>	
<b>38 SODIUM BICARBONATE TEST</b> .....	68
<i>(strong organic acids)</i>	
<b>39 TEST FOR OBTAINING ESTERS</b> .....	69
<i>(carboxylic acids, anhydrides, acid chlorides)</i>	
<b>40 DETERMINATION OF ESTERS BY HYDROLYSIS</b> .....	70
<i>(esters, organic acids)</i>	
<b>41 HYDROXAMIC TEST</b> .....	71
<i>(esters, anhydrides, nitriles, amides)</i>	
<b>42 MODIFIED HYDROXAMIC TEST</b> .....	73
<i>(aromatic sulphonic acids and their chlorides)</i>	
<b>43 HYDROLYSIS WITH NaOH SOLUTION</b> .....	75
<i>(amides, nitriles)</i>	
<b>44 FUSSION WITH SODIUM HYDROXIDE</b> .....	76
<i>(sulphonamides)</i>	
<b>45 HINSBERG TEST</b> .....	77
<i>(difference of amines 1°, 2°, 3°)</i>	

<b>46</b>	<b>NITROUS ACID TEST</b> .....	79
	<i>(amines)</i>	
<b>47</b>	<b>NINHYDRIN TEST</b> .....	81
	<i>(amino acids and ammonium salts, primary amines)</i>	
<b>48</b>	<b>COPPER(II) SULPHATE SOLUTION TEST</b> .....	82
	<i>(aminoacids)</i>	
<b>49</b>	<b>TEST ON OXIDATION OF IRON(III) HYDROXIDE</b> .....	83
	<i>(nitro compounds)</i>	
<b>50</b>	<b>REDUCTION WITH ZINC AND AMMONIUM CHLORIDE</b> .....	84
	<i>(aromatic and tertiary aliphatic nitro compounds and nitroso-, azoxy- and azo-compounds)</i>	
<b>51</b>	<b>KONOVALOV TEST</b> .....	85
	<i>(aliphatic nitro compounds)</i>	
<b>52</b>	<b>NITROUS ACID TEST ON NITRO COMPOUNDS</b> .....	86
	<i>(aliphatic nitro compounds)</i>	
<b>53</b>	<b>SODIUM HYDROXIDE TEST</b> .....	87
	<i>(aromatic nitro compounds)</i>	
<b>54</b>	<b>BIURET TEST</b> .....	88
	<i>(peptides, proteins)</i>	
<b>55</b>	<b>PROTEIN PRECIPITATION TEST</b> .....	89
	<i>(proteins)</i>	
	<b>INDEX OF THE MOST IMPORTANT REAGENTS USED</b> .....	91
	<b>INDEX OF THE MOST IMPORTANT EQUIPMENT USED</b> .....	92
	<b>FINAL REPORT (template)</b> .....	93
	<b>COMPLEMENTARY LITERATURE</b> .....	95



## 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*

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# INTRODUCTION

The identification of organic compounds belongs to the primary activities not only of 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  $^1\text{H}$ ,  $^{13}\text{C}$  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)  $\text{PhNH}_2 / \text{PhNO}_2$ , b)  $\text{Et}_2\text{O} / \text{EtOAc}$ , c)  $\text{PhNO}_2 / \text{CH}_3(\text{CO})\text{Ph}$ ; purification of solids by crystallization and sublimation of a) 1-Br,4- $\text{NO}_2\text{-C}_6\text{H}_4$ , 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  $^{13}\text{C}$ ,  $^1\text{H}$ ; 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.