

**LABORATORY EXPERIMENTS  
ON THE CLASS REACTIONS  
AND IDENTIFICATION OF  
ORGANIC SUBSTANCES**

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Laboratory experiments on the class reactions and identification of organic substances by  
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**SAMUEL P. MULLIKEN & ARTHUR A. NOYES**

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## PREFACE

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THIS collection of laboratory experiments in organic chemistry has been prepared especially for the use of the classes of the Massachusetts Institute of Technology, as a supplement to the ordinary course of instruction in preparation work. The authors' experience has shown that the preparation of typical organic substances in accordance with the plans followed in the manuals of Gattermann, Levy, Fischer, etc., teaches satisfactorily the manipulative methods of organic chemistry and the manner of execution of the leading synthetic processes, but that it fails, to a surprising extent in the case of most students, to give a knowledge of the important characteristics of the various classes of organic compounds, and therefore of the fundamental principles of the science. Unless the instructor is continually on the alert, the course of preparation work becomes almost unavoidably a routine following of directions.

Although the primary purpose of the experiments here described is to illustrate the characteristic reactions of organic compounds, their analytical significance is a feature of no slight importance; for, both in research and technical work, the chemist has frequent occasion to identify the substances he meets with. On this account, and also because it is always desirable to make evident to the student some practical use of the information presented to him, the analytical side of the experiments has been made prominent; and an important part of the course consists in the identification of unknown compounds and the quantitative separation of mixtures by methods devised by the student himself with the help of the knowledge gained from the experiments with known substances.

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4 *PREFACE.*

It is believed that the entire omission of explanatory statements of what occurs in the experiments will cultivate the student's power of observation, and cause him to consider more carefully the principle illustrated; while the work on the identification and separation of unknown substances will afford abundant opportunity for original thought.—It is assumed that a brief course of lectures on the outlines of organic chemistry has preceded the laboratory experiments.

Owing to the great importance, in the opinion of the authors, of instruction of this kind, and owing to the fact that no text-book presenting it exists, it has not seemed advisable to postpone the publication of the general plan, although it is undoubtedly imperfect in many matters of detail.

The authors desire to express their indebtedness to Dr. J. F. Norris for many valuable suggestions, and to Messrs. H. M. Loomis, A. P. Norris, and C. L. W. Pettee, for their investigations on the applicability of many of the important tests.

## PART I.

### EXPERIMENTS ILLUSTRATING THE CLASS REACTIONS OF ORGANIC COMPOUNDS.

#### *Introductory Explanations and Directions.*

THE following experiments serve to illustrate some of the most important properties and reactions of the various classes of organic compounds. The results obtained are to be regarded as characteristic of the whole class as defined by the heading above the experiment. It must, however, be clearly understood that all compounds of the class do not give these reactions with equal facility—that, indeed, complex members of the class, and especially those belonging at the same time to two or more classes, often do not give them at all. Moreover, many of the reactions are general for more than one class; but, when this is true, it will be pointed out, as far as practicable, in the notes following the experiments.

Statements of what occurs are omitted from the following directions; and the student is expected to observe carefully and record fully in the note-book everything that happens, even those things of apparently minor importance. Attention should be directed not only to what may be seen, but also to any odor or to any heat effect developed. The observed phenomena should then be fully explained with reference to the new compounds formed; and all the reactions where known products are formed should be written,



using structural formulæ. Works on organic chemistry should be consulted when necessary. The notes following the experiments must be very carefully studied.

After completing the experiments, make a table in the note-book showing, as far as possible, the behavior of each class of compounds towards each of the following reagents: cold dilute alkaline hydroxides; boiling concentrated alkaline hydroxides; concentrated sulphuric acid alone and with subsequent addition of water; sodium; and bromine water.

Students are warned that many of the reactions may take place suddenly and with great violence, and that, therefore, cautious manipulation is necessary, particularly in test-tube experiments.

*Behavior of Organic Substances on Ignition.*

1. Ignite in a small crucible, as long as any change occurs, first a little benzoic acid, and then a little starch.

Repeat the experiment with a little anhydrous sodium acetate. Add a drop of dilute hydrochloric acid to the residue after cooling.

This reaction with hydrochloric acid is given only by organic salts of the alkalis and alkaline earths.

*Detection of Water in Organic Liquids.*

2. Add 0.2 gram of fused pulverized potassium carbonate to 5 cc. of common 95 per cent. alcohol. Shake and set aside for an hour.

Repeat this experiment, using 5 cc. of the same alcohol diluted with 1 cc. of water.

3. Add 0.5 gram copper sulphate, freshly dehydrated by ignition at a low temperature, to 5 cc. of

ordinary 95 per cent. alcohol. Shake and allow the mixture to stand for about one hour.

*Reactions Distinguishing Double and Triple-Bonded from Single-Bonded Compounds.*

4a. Dissolve 0.5 gram of amylene in 5 cc. of carbon tetrachloride, and add gradually a 10 per cent. solution of bromine in carbon tetrachloride as long as any action occurs.

Repeat this experiment, using first cinnamic acid, then phenol, and finally toluene, in place of the amylene. In the cases of the cinnamic acid and toluene, after allowing the mixture to stand in the cold for two or three minutes, heat it to boiling for about a minute.

Decolorization without evolution of hydrobromic acid shows *addition*, and therefore the probable presence of a double or triple bond. Decolorization accompanied by evolution of hydrobromic acid shows *substitution*; but it does not necessarily exclude the possibility of a simultaneous addition, which may even be inferred in case the evolution of hydrobromic acid is not proportionate to the rate of decolorization. If even on heating no action occurs, or if only a slow action accompanied by evolution of hydrobromic acid takes place, it is probable that no double or triple bond is present; it is true, nevertheless, that there are some double-bonded compounds (for example, fumaric, maleic, and the nitro-cinnamic acids) which form addition-products only very slowly or not at all under the conditions of this experiment. On the other hand, amines, phenols, and most aldehydes and ketones, like double-bonded compounds, decolorize the bromine solution instantly; in the case of amines, often without evolution of hydrobromic acid.

4b. Dissolve 0.5 cc. of allyl alcohol in 5 cc. of water, and gradually add bromine water as long as decolorization takes place.

Repeat the experiment, using ethyl alcohol instead of allyl alcohol.

As bromine water is used as a reagent in other cases, this experiment is introduced here, in order to show its behavior with unsaturated compounds. But, as a means of distinguishing them from saturated bodies, the test in carbon tetrachloride is far more satisfactory: for, when water is used as the solvent, any hydrobromic acid formed is absorbed by it, so that it is not readily possible to distinguish substitution from addition; moreover, besides these two actions, oxidation often takes place in aqueous solution; and finally, the unsaturated compounds which fail to react in carbon tetrachloride are almost equally inert in water, so that the latter solvent has no advantage in this respect.

5. Add 0.2 gram of cinnamic acid to 5 cc. of sodium carbonate solution, and then add drop by drop about 1 cc. of a one per cent. solution of potassium permanganate.

Repeat the experiment, using first amylene and then toluene in place of the cinnamic acid.

The oxidation takes place almost instantaneously with unsaturated compounds, and with some saturated ones, such as formic acid, malonic ether, phenols, oxybenzoic acids, benzaldehyde, acetone, acetophenone, glycerine, and some sugars. But most saturated compounds are oxidized much more slowly, if at all.

*Reactions of Triple-Bonded Compounds Containing the ( $\equiv C - H$ ) Group.*

6. Add 1 cc. of ammoniacal cuprous chloride solution to 10 cc. of a saturated aqueous solution of acetylene and shake.

*Behavior Distinguishing Saturated Fatty Compounds Containing No Complex Alkyl Radicals from Other Compounds.*

7. Roll a piece of fine copper gauze 1 cm. square around the end of a copper wire. Dip this in succes-