A COURSE OF PRACTICAL CHEMISTRY FOR AGRICULTURAL STUDENTS. VOLUME II. PART I

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A Course of Practical Chemistry for Agricultural Students. Volume II. Part I by H. A. D. Neville & L. F. Newman

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H. A. D. NEVILLE & L. F. NEWMAN

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PREFACE

THE following exercises in Practical Agricultural Chemistry are designed primarily for the use of students taking the course for the Degree in Agricultural Science at Cambridge University. Volume I is intended to cover the first year's course on the Chemistry and Physics of the Soil, while Volume II covers the second year's course on the Chemistry of Foods. The latter is divided into two parts: Part I dealing with such exercises in Pure Organic Chemistry as are essential for agricultural students, and is entirely qualitative, while Part II deals with quantitative estimations and technical analyses of food stuffs. This division has been considered desirable, as students who have already taken a course in Organic Chemistry will find it unnecessary to work through Part I of Volume II. Volume III deals with the special advanced Chemistry of Insecticides and Fungicides. Food Preservatives, etc. It is hoped that this division into convenient booklets will make them useful to teachers. The exercises are designed to illustrate most of the essential points in Agricultural Chemistry and to require the minimum of apparatus.

A Course of Practical Work was compiled by Professor T. B. Wood some years ago for the use of his classes, and this forms the foundation of a portion of the present exercises. The sincere thanks of the authors are due to Professor Wood and the Syndics of the University Press for their permission to utilise that material.

In addition to the directions for practical work a series of notes has been added to each day's work to explain just the points which considerable experience in teaching in a laboratory has shown to be those on which the student usually needs assistance from the demonstrator to explain the reason and reactions of the experiments as they are being carried out.

It is fully recognised that many of the experiments and accompanying notes could have been elaborated with advantage, but the necessity of restricting apparatus to the limits which it is possible to supply for the use of large classes, and the fact that the notes are only intended as laboratory guides, and not to supersede lectures, have determined the present scope of the volumes.

H. A. D. N. L. F. N.

1 October 1919.

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COMPOSITION OF ORGANIC COMPOUNDS

1. In a small porcelain crucible ignite in turn small quantities of (a) sugar, (b) starch and (c) naphthalene. Note that in all three cases the substance chars at first and afterwards burns away completely, leaving no residue. (See note a.)

2. Repeat Expt. 1 using this time small quantities of (a) casein, (b) hay and (c) oil cake. Note that in these cases, while the greater portion of the material burns away, a small quantity of ash is left behind. (See note a.)

3. Well mix a small quantity of sugar with copper oxide and place the mixture in a dry test-tube. Fit the test-tube with a cork and a small leading tube bent once at right angles. Holding the test-tube obliquely, place a small quantity of anhydrous copper sulphate in the upper part of the tube near the cork and, having replaced the cork and leading tube, allow the latter to dip into lime-water contained in a second test-tube. With the tubes in the position indicated, strongly heat the mixture of sugar and copper oxide. Note that water is formed and collects in the cooler parts of the first test-tube, where it turns the white anhydrous copper sulphate blue, while, at the same time, the lime-water in the second test-tube turns milky. (See note b.)

4. Boil with dilute caustic soda solution in a test-tube small quantities of (a) oxamide and (b) case in. Test the steam evolved with red litmus paper and by the smell, and note that while ammonia is evolved from the oxamide, no ammonia is evolved from the case in. (See note c.)

5. Repeat Expt. 4 but instead of using caustic soda solution use dry powdered soda-lime intimately mixed with the organic substance. Note that in this case ammonia is evolved from both substances. (See note c.)

6. In a small test-tube heat up a small quantity of casein with two or three very small pieces of metallic sodium. When all reaction has finished, remove the test-tube from the flame, allow to cool alightly but, while still hot, drop the test-tube into cold water in a small porcelain dish. (Stand well back from the dish while this portion of the experiment is being done.) The test-tube on touching the cold water will crack and is then broken up into small pieces with the help of a rod, so that the water comes into contact with all the charred mixture in the tube. Filter the solution in the dish and divide into two portions. Saving one portion for Expt. 7, treat the other portion of the filtrate as follows. Add a few drops of ferrous sulphate solution 6

and a few drops of ferric chloride solution and boil. Cool and acidify with dilute hydrochloric acid. Note the greenish-blue or blue coloration or precipitate produced. (See note d.)

7. Using the second portion of the filtrate obtained in Expt. 6, divide it into three parts and carry out the following tests:

(a) Place a small quantity of the first part on a clean silver coin and note the black stain produced on standing.

(b) To the second part add acetic acid until acid and then lead acetate solution. Note the black precipitate produced.

(c) To the third part add a few drops of sodium nitroprusside solution and note the violet coloration produced. (See note e.)

8. In a porcelain crucible place a small quantity of a mixture of equal portions of potassium carbonate and potassium nitrate. Heat the mixture over the bunsen flame until fused and add to the fused mass, in small portions at a time, a small quantity of casein. When all the casein has been added, allow the mass to cool and dissolve out the mixture from the crucible by boiling crucible and contents in a beaker containing enough water to just cover the crucible. Filter the solution obtained and test the filtrate for sulphate and phosphate by the methods described in Volume I. Note the presence of both sulphate and phosphate in the liquid. (See note f.)

NOTES

Note a. Most pure organic bodies burn away completely on being ignited strongly. In the first stages of the ignition a black residue of carbon may be produced, but further ignition will remove this as the carbon is oxidized to carbon dioxide. Naturally occurring organic substances will, in almost all cases, leave a varying amount of ash and examples of this are the substances mentioned in Expt. 2. This ash is generally an impurity but in a few cases may be an essential constituent of the organic body. An extreme case of this is where a metallic salt of an organic acid is under examination. Here the amount of ash may be quite large and will generally consist of the oxide or carbonate of the metal concerned.

Note b. In this experiment the sugar on ignition reacts with the copper oxide. The hydrogen of the sugar forms water and the carbon, carbon dioxide, the necessary oxygen supplied by the copper oxide which breaks up leaving metallic copper. The production of water is demonstrated by the white anhydrous copper sulphate $CuSO_4$ taking up water and passing to the blue hydrated salt $CuSO_4 5H_3O$. The carbon dioxide produced reacts with the lime-water to form calcium carbonate as explained in Volume I.

Note c. While a few organic compounds containing nitrogen, of which examide is an example, liberate nitrogen as ammonia on boiling with aqueous soda, the greater number do not, and the presence of nitrogen can only be shown by strongly igniting the substance with soda-lime. Nearly all organic bodies of agricultural importance give off their nitrogen in the form of ammonia when heated with soda-lime. This test is not of universal application and the only satisfactory general test is the one described in Expt. 6.

Note d. This experiment depends on the production of sodium cyanide formed by the union of the sodium with the carbon and hydrogen in the organic substance. The cyanide so formed reacts with the ferrous hydroxide, formed when the ferrous sulphate is added to the alkaline solution, to give sodium ferro-cyanide which in turn reacts with the ferric chloride to give ferric-ferro-cyanide which is Prussian blue.

 $\begin{array}{l} \textbf{FeSO}_4 + 2\textbf{NaOH} = \textbf{Fe}(\textbf{OH})_2 + \textbf{Na_2SO}_4\\ \textbf{Fe}(\textbf{OH})_2 + 6\textbf{NaCN} = \textbf{Na_4Fe}(\textbf{CN})_6 + 2\textbf{NaOH}\\ \textbf{3Na_4Fe}(\textbf{CN})_6 + 2\textbf{Fe}_2\textbf{Cl}_6 = \textbf{Fe}_4(\textbf{Fe}(\textbf{CN})_6)_3 + 12\textbf{NaCl}\\ (\textbf{Prussian blue}) \end{array}$

The depth of the colour produced will depend on the amount of nitrogen present. A very small quantity of nitrogen frequently gives only a greenish-blue coloration, a larger quantity a blue coloration, while a considerable quantity gives a blue precipitate.

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Note e. On fusing with sodium any sulphur contained in an organic compound is converted into sodium sulphide. This will react with silver to form black silver sulphide, Ag₃S, with lead acetate to form black lead sulphide, PbS, and with sodium nitroprusside to give a purple colouring matter. The last-named test is particularly delicate.

Note f. An organic body containing sulphur or phosphorus reacts with the fused salts. The sulphur is oxidized to sulphate and the phosphorus to phosphate. These are then tested for in the ordinary way.

General note. The presence of halogens (= chlorine, bromine, iodine or fluorine) in organic compounds can be shown by heating the substance with metallic sodium as in the test for nitrogen and testing the solution for chlorides, bromides, etc. As however halogens containing organic substances are not often met with in agricultural chemistry, this experiment is not described in detail.

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