Development of New Indirect Spectrophotometric Methods of Analysis Using Phenolphthalin as Reagent

A Thesis Submitted by

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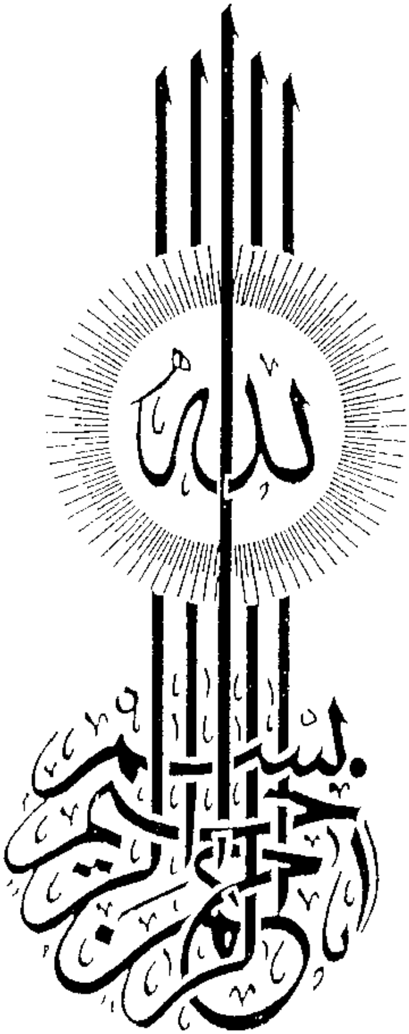
TO MY FAMILY


بسم الله الرحمن الرحيم

Development of New Indirect Spectrophotometric Methods of Analysis Using Phenolphthalin as Reagent

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Note

Most of the work presented in this thesis has been published in the following papers:

1. Salah A. Shahine and Rafaa M. Mahmoud, Indirect Spectrophotometric Determination of Some Reducing Agents Using Phenolphthalin as Reagent, Mikrochim. Acta, 2, 431-434 (1978).

2. Salah A. Shahine and Rafaa M. Mahmoud, Indirect Spectrophotometric Determination of Reducing Sugars and Ascorbic Acid Using Phenolphthalin as Reagent, Mikrochim. Acta, 1, 119-122 (1980).

3. Salah A. Shahine and Rafaa M. Mahmoud, Selective Spectrophotometric Determination of Gold Using Phenolphthalin as Reagent, Bulletin of the Faculty of Engineering, Ain Shams University, 11, P. Chem., 1/1-1/7 (1980).

Besides, the following article is in preparation: Salah A. Shahine and Rafaa M. Mahmoud, Rapid Spectrophotometric Determination of Traces of Some Anions in Water.

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Rapid Spectrophotometric Determination of Traces of Some Anions in Water Using Phenolphthalin as Reagent

SUMMARY

The work presented in this thesis deals with the use of phenolphthalin (reduced form of phenolphthalein) for developing new indirect spectrophotometric methods of analysis. The colourless alkaline solution of phenolphthalin turns red by the action of oxidising agents that can act in alkaline media (due to oxidative regeneration of phenolphthalein.

The first chapter of this thesis contains/a detailed survey of the literature dealing with the direct and indirect colorimetric methods of analysis, the organic oxidising and reducing agents used in this type of analysis and the use of phenolphthalin as an oxidation-reduction reagent./

Chapter II comprises/an indirect method for the spectrophotometric determination of microgram amounts of quinol, hydroxylamine and hydrazine. A known excess of potassium ferricyanide solution is added to the sample solution to oxidise the compound to be determined; the excess of ferricyanide is then determined spectrophotometricaly using phenolphthalin./

An indirect spectrophotometric method for the determination of reducing sugars and ascorbic acid is described in chapter III. The method is based on the reaction of an excess of ferricyanide with the reductant and the determination of the surplus ferricyanide spectrophotometrically using phenolphthalin.

A modified method for the indirect spectrophotometric determination of gold using phenolphthalin as a reagent was investigated in chapter IV. In this method, gold is extracted from other metals as bromoauric acid, recovered in water and determined spectrophotometrically by oxidising the colourless phenolphthalin to the red phenolphthalein.

Chapter V describes a new indirect spectrophotometric method for the determination of bromide, iodide, thiocyanate, sulphide and cyanide in water. The method is based on the exchange reaction with silver ferricyanide and the subsequent spectrophotometric determination of the displaced ferricyanide using phenolphthalin.

All the reactions involved in the present work were critically studied and the results were analyzed statistically.

CHAPTER I

General Introduction

CHAPTER I

DIRECT AND INDIRECT COLORIMETRIC METHODS

Colorimetric methods can generally be classified into direct and indirect methods depending on the way by which the coloured product, to be measured, is formed. A survey of some of these methods covering the various kinds of reaction is presented in the following pages.

Direct Colorimetric Methods:

In this type, the constituent to be determined is transformed into a coloured compound by one of the following methods:

1. The constituent is allowed to react with a reagent with which it forms a stable coloured complex. Examples: calcium with naphthalhydroxamate (1), palladium with triphenylphosphine (2), ruthenium with isonitrosobenzoylacetone (3), beryllium with Chrome-Azurol S (4), titanium with diantipyrinylmethane and tartaric acid (5), cobalt with 2-nitroso-1-naphthol (6), thorium with galangin (3,5,7-trihydroxyflavone) (7), copper(II) and mercury(II) with di-1-naphthylthiocarbazone (8), ferric with thiocyanate (9) or with chelidamic acid [4-hydroxypyridine-2,6-dicarboxylic

acid] (10), ferrous with 1,3-dimethyl-5-nitroso-2-thioxoperhydropyrimidine-4,6-dione (11), tungsten(VI) by forming tungsten-catechol violet-hexadecylpyridinium (1:1:2) complex (12), osmium(III) with oxalic acid (13), nitrate with rhenium and furil α-dioxime (14), iodide with butylrhodamine S (15), and aliphatic [alcoholic] hydroxyl in the range C_8 to C_{18} with vanadium 8-hydroxyquinolinate (16). The latter method was modified to determine methanol in aqueous and non-aqueous solutions (17).

2. The constituent is oxidised to a higher state which has a characteristic colour. Examples: manganese(II) is oxidised to permanganate (18), chromium(III) to dichromate (19), and ethanol to an absorbing species (20).

3. The constituent is reduced to a lower oxidation state having a characteristic colour. Examples: molybdenum (VI) is reduced to a lower oxidation state which has a blue colour (21), and gold(III) to a purple colloidal gold (22).

Other miscellaneous direct methods have been based on the formation of heteropoly-acids; for example, vanadium is determined as 5-molybdovanadic acid (23). Few methods are based on the nitration of the reagent; for example, nitrate by nitration of 2,6-xylenol (24). Others