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Micro Analysis of Medicinal Compounds using Ammonium Metavanadate Reagent: Study of Alcohols and Sugars

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Abstract: *The current study depicts that the determination of butane 1,3 diol, butane 2,3 diol, triethylene glycol, methanol, ethanol, n-propanol, n-butanol, 3-pentanol, propane 1,2 diol,and tetraethylene glycol with the use of V(v) reagent. The study also shows that the oxidizing capacity of ammonium metavanadate (v) with certain organic compounds, the oxidation of certain sugars was studied using an accurate and quantitative procedure has been described for the determination of certain mono and disaccharides on micro scale.*

Background*: Drugs are substances or products with a definite physiological activity that are used or intended to be used to modify or explore physiological systems or pathological states for the benefit of the recipient. It is used in the prevention, diagnosis, treatment or cure of diseases in man or other animals. The determination of these biologically active functional groups is important for the quantitative determination of drugs. Physiological activity of a compound is associated with particular structural unit or particular functional groups. These functional groups are biologically active.*

Materials and Methods*: Aliquots containing 1 to 5 mg of glucose were taken in flask and 1 ml of 0.3 N, V(v) reagent and 5 ml of 10 N sulphuric acid was added to it. The reaction was carried out on a boiling water bath for 5, 10, 15, 20, 25, 30, 35, 40, 45 minutes. After the prescribed reaction time contents were cooled at room temperature and the unconsumed V(v) reagent was determined. The unconsumed V(v) reagent was titrated against 0.025 N ferrous I ammonium sulphate using N-phenyl anthranilic acid as an indicator. A blank experiment was also run under identical condition, using all the reagents except the sample. The recovery of the sample was calculated.*

Results*: In the view of the reactions discussed both sugars and alcohols were studied using the reagent. It has been found that sugars containing aldehydic group are oxidised to formic acid only. In case of Ketonic sugars also formic acid is the end product but ketonic group appear to be oxidized to carbon dioxide. Polymers of Ethylene glycol like triethylene glycol and tetra ethylene glycol consumes 8 equivalent of V(v) reagent to give corresponding dicarboxylic acids.*

Conclusion: *The current study depicts that the determination of butane 1,3 diol, butane 2,3 diol, triethylene glycol, methanol, ethanol, n-propanol, n-butanol, 3-pentanol, propane 1,2 diol,and tetraethylene glycol with the use of V(v) reagent. The study also shows that the oxidizing capacity of ammonium metavanadate (v) with certain organic compounds, the oxidation of certain sugars was studied using an accurate and quantitative procedure has been described for the determination of certain mono and disaccharides on micro scale.*

Key Word*: Medicinal Compounds,Alcohols, Sugars, Ammonium Metavanadate Reagent.*

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I. Introduction

Drugs are substances or products with a definite physiological activity that are used or intended to be used to modify or explore physiological systems or pathological states for the benefit of the recipient. Itis used in the prevention, diagnosis, treatment or cure of diseases in man or other animals. The determination of these biologically active functional groups is important for the quantitative determination of drugs.Physiological activity of a compound is associated with particular structural unit or particular functional groups. These functional groups are biologically active.

Lindberg and Missionry[1,2] employed sodium borohydride as the reductant for determining carbohydrates. The sample was dissolved in water and treated with solution of sodium borohydride. After the reaction sis completed the residual borohydride is determined by measuring the hydrogen evolved upon the addition of acid. Peat, Whelan and Roberts' determined the degree of polymerization of reducing oligosaccharides, by means of sodium borohydride.

Various other titrimetric method[23-25] have been proposed from time to time for the determination of alcohols. Dusic[26] determined diols by using $NaIO_4$ solution as oxidant excess 10, was determined potentiometrically with $N_2H_4H_2S0$, at platinum electrode. Singh et al oxidised methanol and glycerol with

excess amount of permanganate in alkaline solution. The excess reagent was titrated potentiometrically with sodium formate in presence of $BaCl_2$. Beck[2] determined ethylene glycol and glycerol with trivalent copper by potentiometric titration. Stognushko et al developed a potentiometric method for the analysis of phosphorylated product at higher alcohols. A suitable method has been suggested for the determination of Crotyl alcohol with chloramine-T by Naidu et al Haring et al[32] evolved a method for the determination of allyl alcohol with manganese pyrophosphatic. Various metal ions[33-34] were also employed for the determination of alcohols. Oxidation of secondary alcohol as well as ally! alcohol[36] with PFC and Ni(iv) respectively are carried out. Various Methods developed for the determination of alcohols, with different inorganic reagents. In the present work a new titrimetric method has been described for the micro determination of some monohydric and dihydric alcohols with the use of ammonium metavanadate as an oxidising reagent. This is quick, easy and accurate procedure and is of general applicability. The accuracy of the method is within +1% is most of the cases.

Komers et al proposed a method for the determination of small amount of MeOH and EtOH in aqueous solution. The method based on combination of stripping and gas chromatography technique. Normal C_7-C_{16} aliphatic alcohols mixture was analysed by gas Chromatography' at 190° on a column packed with 10% polyethyleneglycol. Other chromatographic techniques like liquid chromatography and gas chromatography were also employed for the determination of monohydric and dihydric alcohols. Glycerol was determined spectrophotometrically in its industrial liquors by measuring the absorbance at 580 nm of the violet colour several other spectrophotometeric method' are also available in the literature for the determination of alcohols. Barkat evolved a titrimetric method using N-bromosuccinimide as oxidant for the determination of primary and secondary alcohols. Kruse et al developed an oxidimetric method for differentiating between three types of saturated alcohols using Nhaloimides.A similar type of method has also been proposed by Severin. It has been claimed that the method is rapid and accurate compared to the methods proposed by Lucas and Ritter. Litter as well as Jones studied the reaction. Kinetics of oxidation of alcohols using pentavalent vanadium as oxidant and excess vanadate was back titrated with Mohr's salt or with Fe (II) sulphate solution.

Colorimetric methods are also available for the determination of sugars. The first procedures were developed by Floin and Wu[4] in 1919 which is based in the conversion of cupric ions to the cuprous sulphate by the presence of reducing sugars. The cuprous ions subsequently reduce phosphotungstic acid to a blue complex which is measured calorimetrically. Dearing' described a micro method for the estimation of cellulose Wahba and Coworkers[6] converted glucose to glucosazone and measured the yellow colour of its solution. Shallenberger and Mooros[7] developed the colour with a reagent containing copper sulphate and arsenomolybdates and measured the solution at 500mg Various phenolic compounds have been recommended for the colorimetric determination of carbohydrates. Dubois[8] used phenol for the determination of reducing sugars. Tillmans[9] determined the reducing sugars with the use of thymol. Sorenson, Fisher and Lindh 10-12 used resorcinol and phloroglucinol for the determination of reducing sugars.

Rebega[38] and Rlbega and coworkers[39] used cuprithiosalicylate as oxidising agent and have determined sucrose, fructose and glucose empirically. Celsi and SarrailW° oxidised glucose, surcose and lactose with Cu(14) in the mixture of K_2CO_3 and KCNS. The precipitated CuSCN was estimated using Fe(III) and Ag(I). Defrates and castles estimated reducing sugars with Fehling's solution using electrometric end point detector. Potassium ferricyanide has been widely used in determining sugar[42, 48]. According to Blom and Rosted[49] this method of estimation so sugars with ferricynide is much inferior to cuprimetry method in as much as ferricyanide procedures, side reactions and it attacks other organic substances present in the sugar. Lately there have been good deal of attempts to regularise the oxidation of sugars with ferricyanide better results have been claimed when sugar is treated against a boiling alkaline Solution of hexacyanoferrate (111)[50].

Hass and Lynch22 developed a method for the determination of carbohydrates. Mayer and Isbe1[23] employed radioactivity and determined end groups in carbohydrates. Browne[24] determined carbohydrates by biological methods. Figueirede[25] have developed an improved complexometric method for the analysis of reducing sugars. Amongst the various oxidimetric methods for the determination of sugars, cuprimetry has been of great use from early times. Divalent copper complexed with tartarate or citrate in alkaline medium known as Fehling's solution[26-29] and Benedict's solution respectively.

II. Material and Methods

The unconsumed V(v) reagent was titrated against 0.025 N ferrous I ammonium sulphate using N-phenyl anthranilic acid as an indicator. A blank experiment was also run under identical condition, using all the reagents except the sample. Recovery of the sample was calculated by following expression.

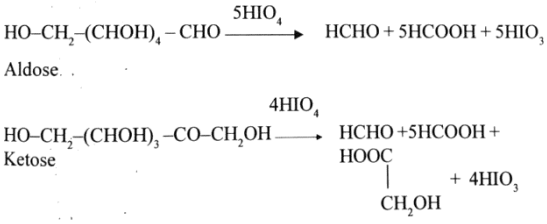
$$\text{mg of the sample} = \frac{M \times N\,(B - S)}{n}$$

Where M = Molecular weight of the sample, N = Normality of Fe(II) solution, B=Volume of Fe(II) consumed to titrate with the blankexperiment, S = Volume of Fe(II) consumed to titrate with the sample experiment and n=Number of moles of V(v) reagent consumed per mole of the sample.

Aliquots containing 1 to 5 mg of glucose were taken inflask and 1 ml of 0.3 N, V(v) reagent and 5 ml of 10 N sulphuric acidwas added to it. The reaction was carried out on a boiling water bathfor 5, 10, 15, 20, 25, 30, 35, 40, 45 minutes. After the prescribed reactiontime contents were cooled at room temperature and the unconsumedV(v) reagent was determined. It was observed that the recovery of thesample become Constant within the reaction time of30 minutes.Keeping the amount of sample reaction time and theconcentration of V(v) reagent as constant. The effect of varyingconcentration of sulphuric acid was studied.

III. Result and Discussion

Various reaction mechanism has been proposed for sugarusing different reagent. Fleury and Co-workers[56] shows that formic acidand formaldehyde are the product when aldoses are oxidized whileglycolic acid is an additional product when ketoses were oxidized with periodic acid.

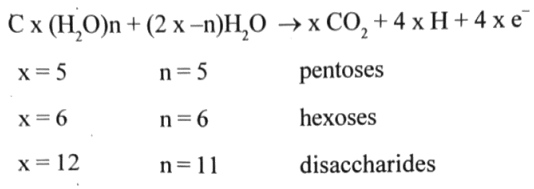


It is evident from the above reaction that varying amounts of periodate are consumed for the two categories of sugars.

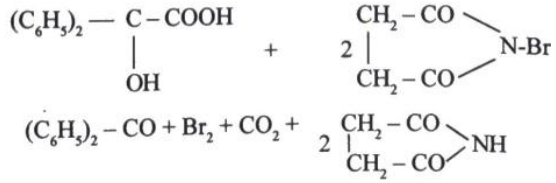
Aldoses are smoothly attacked by hypoiodite and hypobromite consuming two equivalents of the oxidants. As early as 1897 Romijh[62] determined aldoses by oxidising with hypoiodite on the basis of $_c$onsumption of two equivalents. He found that ketoses are not attacked by hypoiodite but methanol, formic acid, glycerine, mannitol and lactic acid interfered in the determination.

Yoshimura and Co-workers illustrated the consumption of 6 equivalents of hypobromite when oxidation was carried out in boiling condition instead of room temperature.

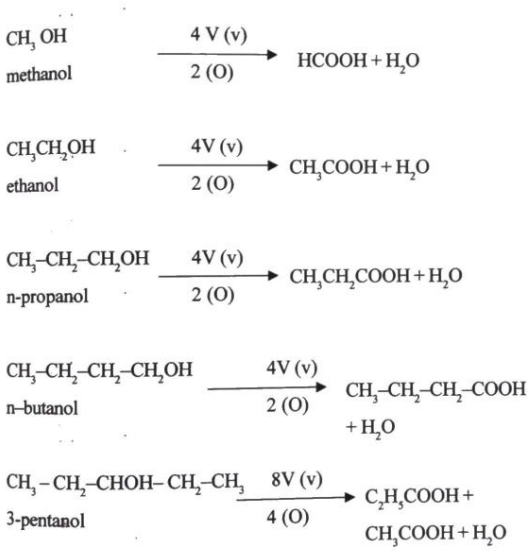
Nasulaev[76] determined glucose with the consumption of two equivalents of IBr. Becke[1-82] standardised Cu(III) against glucose at room temperature assuming the consumption of 8 equivalents of Cu(111) per mole of the glucose.



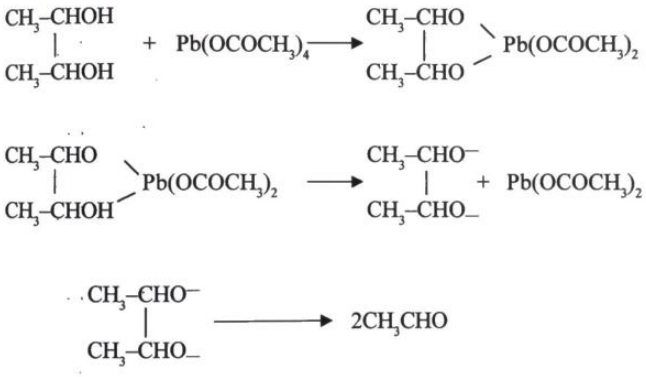
The effect of temperature as mentioned earlier was also studied I and it was noticed that 40°C is sufficient for quantitative oxidation. Beyond 50°C inaccurate results are obtained and a sharp end points not obtained. The stoichiometry of the reaction was also established for each compound are equivalent of methanol, ethanol, n-propanol, nbutanol, 3-pentanol, propane, 1,2 diol, butane 1-3 diol, butane 2,3 diol, triethylene glycol & tetraethylene glycol consumes 4, 4, 4,4, 8, 6 , 12, 6, 8 and 8 equivalent of V(v) reagent respectively. It is well known that primary and secondary alcohols are oxidised to aldehydes and ketones respectively by the use of a mixture



In view of above oxidation reaction and considering the amount I of $_V$ (v) consumed per mole of the primary and secondary alcohols the f$_o$ll$_o$wing course of reaction may be proposed for the oxidation of methanol, ethanol, n-propanol, n-butanol & 3-pentanol with the use of V (v) reagent.



In the above oxidation reaction primary alcohols after oxidation with V (v) gives corresponding carboxylic acid and secondary alcohol produces a mixture of acids. Aldehydes and Ketones are produced in case of 1, 2-glycol as well with certain reagents, notably periodic acid and lead tetraacetate. The reaction affords one of the few ways in which a carbon - carbon single bond can be cleaved readily various theory have been proposed to explain the cleavage, one of which postulates that the glycol is changed to 1, 4-Zwitter ions, which is turn breaks down to form two carboxyl groups. According to this theory the cleavage of 2,3 butane diol with lead tetraacetate[48] may be depicted as follows.



IV. Conclusion

The current study depicts that the determination of butane 1,3 diol, butane 2,3 diol, triethylene glycol, methanol, ethanol, n-propanol, n-butanol, 3-pentanol, propane 1,2 diol,and tetraethylene glycol with the use of V(v) reagent. The study also shows that the oxidizing capacity of ammonium metavanadate (v) with certain organic compounds, the oxidation of certain sugars was studied using an accurate and quantitative procedure has been described for the determination of certain mono and disaccharides on micro scale.

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