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Development of a new method for Micro determination of Carbonyl Compounds

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Abstract: ***A survey of chemical literature of the eighteenth and nineteenth centuries would reveal that major works have been done in the analytical chemistry. Thus it may not be inappropriate to regard analytical chemistry as the oldest field in the broad spectrum of chemical analysis. The foundation of micro-technique, especially in the field of quantitative organic analysis was laid in the first two decades of present century by Pregl. The sample weights used in methods are in the following increasing order. Macro methods, semi micro method, micro method, sub micro method. For a single run of experiment the sample range for macro method is about 0.5g or more, semi micro method is 50-200 mg. micro method is 1-20 mg and sub micro method is 30-50 mg. the semi micro, micro and sub micro methods of analysis possesses advantage of being economical, fast, accurate and convenient. Sub micro method employ very small amount of the sample having considerable importance for biochemical analysis and for the study of substances available in very minute quantities as in the drugs and other pharmacologically active substance. In the same process Carbonyl Compound, we can determine to development of new method of Carbonyl Compounds.***

Drugs are organic substances with a definite physiological activity used in the prevention, diagnosis, treatment or cure of diseases in man or other animals. Physiological activity of a compound is associated with particular structural unit or particular functional groups. These functional groups are biologically active. The determination of these biologically active functional groups is important for the quantitative determination of drugs.

Key Words: : survey, chemical literature, eighteenth, nineteenth, analytical, chemistry, biologically.

Introduction- Analytical chemistry may be defined as the science and art of determining the compounds which they contain. The first quantitative analysis was gravimetric. Robert Boyles styled the process of analysis in 1661. Nearly a century later Bergmann complied and arranged the systematic and methodical way of analysis and this laid a foundation upon which Berzelius and Boss build a system of quantitative analysis. Nearly any physical poetry characteristic of a particular element or compound can be mad the basis of method for its analysis. Analyses of organic compounds via the reaction of their functional groups are more fundamental and elemental analysis because their characteristic properties arise from the functional groups presents in them.

Analytical chemistry involve the three 'S' of selectivity, sensitivity and speed, which spell the ultimate aim of the method of chemical analysis.

Selectivity means the ability to detect and determine one substance in presence of other substances which might be expected to interfere with its identification and determination. Sensitivity means tendency to take maximum accurate results in analytical studies and speed for a reaction vary in different situation.

Oxidative procedure with V(v) reactions are simple and a close control of the variables such as temperature and acid concentrations does not appear to be critical. The preparation of the reagent and its excellent stability warrant further consideration of the applicability in the analysis of organic compounds. For development of new method for micro determination of Carbonyl Compounds.

The compounds containing carbonyl grouping may be either aldehydic of ketonic and both of these could be determined by applying some

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important reactions of the carbonyl function like oxidation, reduction and condensation with the help of reagents. In view of the fact that a large number of naturally occurring compounds contain this carbonyl function and quite a few synthetic or naturally occurring compounds are of practical and industrial importance, the determination of the carbonyl function or of the compounds containing this group is of special significance.

A detailed survey of literature reveals that earlier analytical methods developed for the purpose were mostly directed towards the determination of individual compounds like acetone or formaldehyde. One such survey was published by Mitchell, which covers references up to 1951. Although some of the methods are gravimetric, it has been found that volumetric or colorimetric method are better and these frequently form the basis of the specific methods.

Methods based on chromatography, spectrophotometry, polarography or mass spectrometry has been proposed from time to time and are frequently employed. Whereas most of the methods originally developed were available on macro scale, they have been modified for operations on the semimicro and micro scale determinations.

When sodium bisulphite reacts with carbonyl compound addition product is formed and method based on this reaction was first employed by Ripper for the determination of formaldehyde. The sample was added to an excess of bisulphite and the unreacted bisulphite was estimated by titration against, iodine. On account of the unstable character of bisulphite, the method gave erroneous result, and consequently Parkinson and Wagner modified this method. They added excess of iodine to the reaction mixture to react quickly with the free bisulphite and the excess of the iodine left was titrated against sodium bisulphite.

Method based on oxidation reaction:

Methods based on oxidation can be applied to the carbonyl compounds which are aldehydic in nature and in the course of oxidation the aldehydic function is oxidized to the carbonyl function. Silver oxide has been employed as an oxidant by several workers like Bailey.

Present work- A survey of literature revealed that ammonium metavandate (v) has not been utilised for the determination of some aldehydic compounds. This encouraged me to study the reaction of ammonium metavandate (v) on aldehydes. A rapid method for the micro determination of aldehydes has thus been evolved.

Approach of the reaction- For testing the quantitative validity of the reaction salicylaldehyde was taken as test sample. Different amount of the sample were allowed to react with varying amounts of V(v) reagent at 40oC for different intervals of reaction time. The stoichiometry of the reaction was established for each samples and as possible course of reaction was suggested.

Effect of reaction time- Keeping amount of salicylaldehyde, concentration of V(v) as constant and the reaction time was varied from 1-45 minuted. The reaction contents were heated at a constant temperature bath maintained at 40oC. At this temperature the reaction is completed within 20 minutes. In case of salicylaldehyde 100% recovery of the sample was obtained within 20 minutes. In case of salicylaldehyde 100% recovery of the sample was obtained within 20 minutes at 40oC.

Table 1

Micro determination of Salicylaldehyde with 0.3 N, V(v)

Aliquots taken (ml)	Amount present (mg)	Reaction time (minutes)	Amount recovered (mg)	Stoichi-ometry	Error (%)	S.D.(mg)	C.V. %
1	1.0000	20	1.0051 1.0065 1.0047	2	+0.51 +0.65 +0.47	0.0094	0.0734
3	3.0000	20	2.9922 3.0084 3.0126	2	-0.76 +0.78 +0.42	0.0107	0.3561
5	5.0000	20	5.0155 5.0180 4.9930	3	+0.31 +0.36 -0.14	0.0137	0.2735
7	7.0000	20	6.9832 6.9825 6.9755	2	-0.24 -0.25 -0.35	0.0042	0.0600
9	9.0000	20	9.0261 9.0342 9.0216	2	+0.79 +0.38 +0.74	0.0063	0.6970

In each case three determinations were done.

Table 2

Micro determination of Pyridine-2-aldehyde with 0.3 N, V(v)

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Aliquots taken (ml)	Amount present (mg)	Reaction time (minutes)	Amount recovered (mg)	Stoichi-ometry	Error (%)	S.D.(mg)	C.V. %
1	1.0000	30	1.0034 1.0020 1.0024	2	+0.34 +0.20 +0.24	0.0070	0.0698
3	3.0000	30	3.0120 3.0075 0.9901	2	-0.40 +0.25 -0.33	0.0115	0.3712
5	5.0000	30	4.9860 5.0070 4.9825	2	-0.28 +0.14 -0.35	0.0132	0.2644
7	7.0000	30	6.9608 6.9720 6.9580	2	-0.56 -0.40 -0.60	0.0074	0.1062
9	9.0000	30	9.0225 8.9811 9.0198	2	+0.25 -0.21 +0.22	0.0231	0.2570

In each case three determinations were done.

Results and Discussions- With the recommended procedure the micro determination of salidylaldehyde, vanillin, o, m and p-nitrobenzaldehyde, p-hydroxybenzaldehyde, veratraldehyde, demethyl amino benzaldehyde, pyridine-2-aldehyde and Thiophene-2-aldehyde was achieved. The result given in (table - 1 & 2). So that the percentage recovery of the sample is fairly constant and accuracy of the method within ±1%.

A survey of literature reveals that the oxidation of aldehydic compounds has been of interesting. The compounds which are aldehydic in nature and in the course of oxidation the aldehydic function is oxidised to the carboxylic function.?

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