

# Study of Trialkylammonium Chromates and 4-Hydroxy-3-Methoxybenzaldehyde (Vanillin) With TAC

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## ABSTRACT

A valuable addition to the prolific oxidant family is the trialkylammonium halochromates ( $R_3NH [CrO_3X]$ ) ( $R = CH_3, C_2H_5, C_3H_7$  and  $C_4H_9$ ,  $X = Cl, F$ ). These reagents are of low cost, readily available and capable of oxidizing numerous organic substrates. A mild and efficient method for the oxidation of diols to the corresponding hydroxyl aldehydes with trialkylammonium fluorochromates ( $R_3NH [CrO_3X]$ ) ( $R = CH_3, C_2H_5, C_3H_7$  and  $C_4H_9$ ) in solution at room temperature, and under microwave radiation was reported by Ghammamy et al.<sup>15</sup>

**Keywords:** Oxidation, TBC, Onium, Chromium.

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## I. INTRODUCTION

Many oxidative reagents have been developed in recent years with some success [1]. In particular, there is continued interest in the development of new chromium (VI) reagents for the effective and selective oxidation of organic substrates, under mild conditions [2]. Significant improvement has been achieved by the use of new oxidizing agents with tetralkylammonium ion like tetrahexylammonium, tetrabutylammonium tetrapropylammonium, tetraethylammonium tetramethylammonium as

counter ions and chlorochromate, fluorochromate, bromochromate and dichromate as oxidants.

With the aim to develop new efficient oxidation protocol, a number of symmetric and asymmetric tetralkylammonium ions with varying alkyl chain length have been synthesized in different research schools to serve as carriers of the oxidants and to deal with organic substrates in organic medium. Some of them have been used in solid state, in solvent free conditions and by microwave irradiation. The effect of tetraalkylammonium ions on the change in water structure is ambiguous. With large alkyl groups and

structuredness of water increase [3-6] while with relatively small alkyl group and more exposed charge on the onium ion, the water structure breaks [7].

## II. ALKYL AMMONIUM IONS AS CARRIERS OF Cr(VI) OXIDANTS

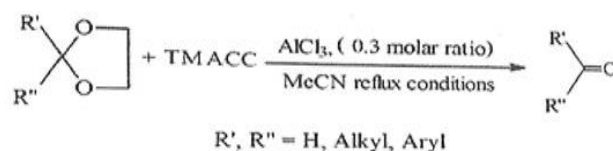
Water-soluble potassium or sodium dichromates are the common laboratory oxidants to oxidize organic substrates and are effective in presence of strong acid. With the advent of organic phase transferring agent, an attempt was made by Sarett School of research, who used pyridine to form salt with  $\text{CrO}_3$ , a Lewis acid, to oxidize some steroidal alcohols in organic solvents [8]. This reagent was subsequently used by other workers without analyzing the structure of the oxidant [9]. Corey, in his novel attempt in establishing pyridinium chlorochromate [8-10] as a versatile oxidant, revisited the Sarett's reagent and discovered it to be pyridinium dichromate [11]. Later on many heterocyclic ammonium ion based Cr(VI) oxidants were synthesized and their oxidation potential towards various substrates were investigated. An extensive review on these oxidants has been published [12].

Recently the kinetics of oxidation of methionine (Met) by TEACC in DMSO leading to the formation of the corresponding sulfoxide was proposed by Patel et al.<sup>11</sup> The reaction was first order each in Met and TEACC and is catalyzed by hydrogen ions. Chouhan et al. reported the oxidation of formic and oxalic acids to yield  $\text{CO}_2$  and aliphatic aldehydes to carboxylic acid by using benzyltriethylammonium chlorochromate (BTEACC) in DMSO. The reagent BTEACC was also used in oxidation of aliphatic primary alcohols to corresponding aldehydes.

Tetramethylammonium fluorochromate (TMAFC:  $(\text{CH}_3)_4\text{N} [\text{CrO}_3]\text{F}$  and chlorochromate (TMACC:  $(\text{CH}_3)_4\text{N} [\text{CrO}_3]\text{Cl}$ ) constitute another class of Cr(VI) oxidants. The reagent were prepared by

the reaction of the corresponding quaternary ammonium salts with  $\text{CrO}_3$  in a 1:1 ratio in acetonitrile medium. TMAFC was used to carry out oxidative deprotection of trimethylsilyl and tetrahydropyranyl ethers or ethylene acetals and ketals to the corresponding carbonyls (Scheme-1). The reagent was also used to oxidize aromatic and aliphatic thiols to corresponding disulfides.

The crystal and molecular structures of TMAFC were determined at 130K by X-ray diffraction. As in case of TEACC in TMAFC also the X-ray data demonstrate inequality between the Cr-O and the Cr-F bonds, which can be attributed to the  $\text{CH}\cdots\text{F}$  hydrogen bond that forms between the methyl hydrogen of the cation and the fluoride atoms of the anion.



**Scheme 1**

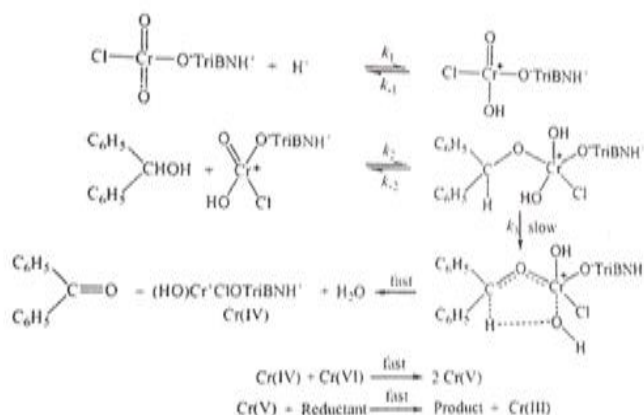
TMAFC can effectively bring oxidation of isopropyl, benzyl, and n-butyl alcohols to corresponding aldehydes. The kinetics of the oxidation reaction was investigated in the presence of p-toluenesulfonic acid. Michaelis-Menten kinetics with respect to alcohols was proposed, demonstrating the quasi-equilibrium formation of an oxidizing agent-alcohol complex. The kinetic isotope effect for benzyl alcohol suggested the cleavage of the C-H bond at the C atom linked to the OH group. A mechanism involving hydride transfer was proposed for the oxidation reaction (Scheme-2). Oxidations of a number of aliphatic, aromatic and allylic thiols to the corresponding disulfide were reported by Imanieh et al. Each reaction proceeds with a two-electron reduction of TMAFC without any detectable amounts of sulphones or sulphonic acids.

### III. TRIALKYLAMMONIUM CHROMATES

A valuable addition to the prolific oxidant family is the trialkylammoniumhalochromates ( $R_3NH [CrO_3X]$ ) ( $R = CH_3, C_2H_5, C_3H_7$  and  $C_4H_9$ ,  $X = Cl, F$ ). These reagents are of low cost, readily available and capable of oxidizing numerous organic substrates. A mild and efficient method for the oxidation of diols to the corresponding hydroxyl aldehydes with trialkylammoniumfluorochromates ( $R_3NH [CrO_3X]$ ) ( $R = CH_3, C_2H_5, C_3H_7$  and  $C_4H_9$ ) in solution at room temperature, and under microwave radiation was reported by Ghammamy et al. [13-17]

Tributylammonium chlorochromate (TriBACC) was used for the oxidation of primary and secondary alcohols in dichloromethane to afford corresponding aldehydes and ketones in high yields. The reagent was prepared by the interaction of tributylamine with  $CrO_3$  and hydrochloric acid in a 1:2:2 mole ratio. Recently, Mansoor et al. Studied the oxidation kinetics of benzhydrols to the corresponding benzophenones by TriBACC. The reaction was first order each in the concentration of TriBACC, benzhydrol and  $H^+$ .

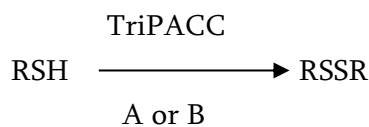
The order of the reactivity for substituted benzhydrol was found to be  $p-OCH_3 > p-CH_3 > p-H >> p-Cl > p-NO_2$  benzhydrol. A mechanism involving hydride transfer was proposed (Scheme 1.14) for the reaction.



**Scheme 2**

Oxidation of alcohols to aldehydes or ketones, anthracene and phenanthrene to anthraquinone and phenanthraquinone respectively were achieved by

tripropylammoniumfluorochromate (TriPAFC). Tripropyl ammonium chlorochromate (TriPACC) was used for oxidative coupling of thiols to corresponding disulfides both in solution and under microwave irradiation (Scheme 3). Both the reagents TriPAFC and TriPACC adsorbed on alumina in solution were also used for the oxidation of thiols to corresponding disulphides.

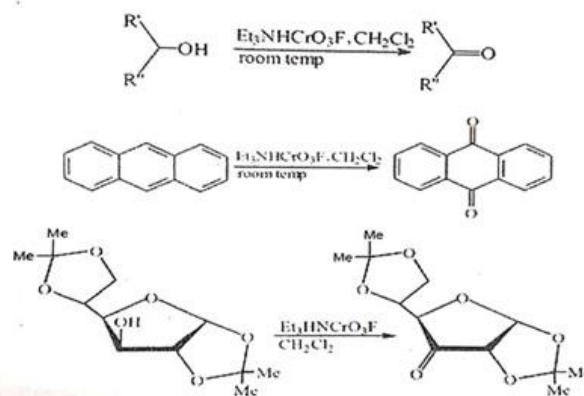


A:  $CH_2Cl_2$  rt

B:  $CH_2Cl_2$ . rt microwave

**Scheme 3**

Triethylammonium fluorochromate ( $Et_3NHCrO_3F$ ) oxidizes primary alcohols, anthracene and naphthalene, and carbohydrates to corresponding oxo derivatives in dichloromethane with high yield (Scheme 4). Conversion of various aliphatic and aromatic thiols into the corresponding disulfides by triethylammonium fluorochromate or triethylammonium chlorochromate supported on silica gel were carried out by Ghammamy et al [14-20].



**Scheme 4**

Trimethylammonium fluorochromate (TriMAFC), which was synthesized from  $CrO_3$ , trimethylamine and aqueous 40% HF in a molar ratio of 1:1:2 was used for oxidation of alcohols to corresponding carbonyl compounds in dichloromethane.

### 3.1 Dialkylammonium Chromates

Diethylammonium chlorochromate (DEACC), a dialkylammonium chromate, was used for the oxidation of primary and secondary alcohols to corresponding carbonyl compounds in aqueous acetic acid medium. The reaction was found to be first order in DEACC and  $H^+$  and followed Michaelis-Menten type kinetics. Chemisorbed on alumina and silica, dimethylammonium chlorochromate (DMACC) was found to be effective for oxidation of alcohols, benzoin and regeneration of carbonyl compounds by oxidative cleavage of C=N under non-aqueous condition.

Sayed-Alangi and his co-workers utilized N-methylbenzylammoniumfluorochromate (MBAFC) and N-ethylbenzylammoniumfluorochromate (EBAFC)<sup>14</sup> for selective oxidation of alcohols to their corresponding carbonyls. The effectiveness of MBAFC and EBAFC was considerably increased upon its adsorption on silica gel. Many functional groups are inert towards this oxidizing agent, including thiols, sulfides and phenols, enhancing the usefulness as chemo selective of these oxidants and the oxidation condition for the synthesis of highly functionalized molecules. The MBAFC and EBAFC were synthesized by treating  $CrO_3$  with aqueous HF and N-methylbenzylamine/N-ethylbenzylamine in the molar ratio of 1:1.5:1.

### 3.2 Alkylammonium chromates

Regeneration of carbonyl compounds from their nitrogen containing derivatives (oximes, p-nitrophenylhydrazones, 4-phenylsemicarbazones and semicarbazone) was achieved using methylammonium chlorochromate adsorbed on silica gel ( $MCC/SiO_2$ ) with good yields. The compound was also used for the oxidation of hydroxyl groups silica to corresponding carbonyl compounds.

The kinetics of oxidation of phenols to quinines by ammonium chlorochromate (ACC) in aqueous acetic acid medium was carried out by Patwari et al. The reaction was first order with

respect to both phenol and ACC and catalyzed by hydrogen ion. The rate of oxidation decreased with increase in dielectric constant of solvent indicating the existence of ion dipole interaction in the oxidation process. The decrease in rate of oxidation with increase in concentration of KCl, was attributed to the formation of a reactive species by interaction of  $Cl^-$  and protonated ACC. Oxidation of some hydrobenzoin to corresponding benzyl by ACC supported on montmorillonite K10 in dichloromethane was reported by Li et al.

## IV. RESULTS AND DISCUSSION

1,2-dihydroxybenzene was first isolated in 1839 by Edgar Hugo Emil Reinsch (1809-1884) by distilling it from the solid tannic preparation catechin, which is the residuum of catechu, the boiled or concentrated juice of Mimosa catechu (*Acacia catechu* L.f.). Upon heating catechin above its decomposition point, a substance that Reinsch first named Brenz-Katechusaure (burned catechu acid) sublimated as a white efflorescence. This was a thermal decomposition product of the flavanols in catechin. In 1841, both Wackenroder and Zwenger independently rediscovered catechol; in reporting on their findings, Philosophical Magazine coined the name pyrocatechin. By 1852, Erdmann realized that catechol was benzene with two oxygen atoms added to it; in 1867, August Kekule realized that catechol was a diol of benzene, so by 1868, catechol was listed as pyrocatechol. In 1879, the Journal of the Chemical Society recommended that catechol be called "catechol" and in the following year, it was listed as such.

### 4.1 Oxidation of 4-Hydroxy-3-methoxybenzaldehyde (Vanillin) with TAC

A few Cr (III) complexes of 4-Hydroxy-3-methoxybenzaldehyde (Vanillin) were synthesized by oxidizing it with ditertiary amyl chromate (TAC) assisted by microwave heating technology. As compared to conventional heating techniques for

chemical synthesis, microwave irradiation proves to be a better technique in terms of reaction efficiency, better yield, and enhanced specificity, less pollution and in achieving energy efficiency. The products were characterized by advanced chemical technological tools, namely, elemental analysis and FTIR spectroscopy. It has been found that the nature and characteristics of the products largely depends on the substrate/oxidant ratio and the solvent used during each set of reaction.

4-Hydroxy-3-methoxybenzaldehyde, i.e., vanillin ( $C_8H_8O_3$ ) is the primary component of the extract of the vanilla bean. Although there has been quite a lot of study on vanillin and its derivatives in the past decade, but the approach towards understanding the chemistry of its reaction with ditertiary amylchromate (TAC) as the oxidant has not been attempted in details. TAC is a versatile reagent used in the oxidation of many organic compounds and also in the synthesis of many metal complexes. But, there are only a few reports on the synthesis and physicochemical studies of chromium (III) complexes of Vanillin. Microwave assisted synthesis<sup>16</sup> is a relatively new technique used by chemists all over the world. The need for implementing green technology in the field of chemical science and research that involves a large use of chemicals for synthetic and analytical processes has shifted our perspective towards using this environment- friendly technique in our work.

However, there are limitations to the instrumentation process and there is a vast scope in improving the technology of microwave ovens commonly used for scientific research so as to use it more efficiently. In view of the above context, we intend to report the microwave assisted synthesis and Infrared (IR) spectroscopic studies of a few compounds obtained by the reaction of vanillin with TAC in different solvent, namely Tetrahydrofuran (THF), dichloromethane and 1,4-dioxane and thereafter present a comparative overview of the

characterization of the products using Fourier Transform Infrared (FTIR) Spectroscopy [20-24].

#### 4.2 Preparation of solutions:

##### Preparation of TAC

TAC, which has been used as the oxidant in the present work was prepared in situ by dissolving a weighed amount of Chromium (VI) oxide in 10 mL tert-amylol.

##### Preparation of reaction mixture

Substrate/oxidant ratio 3:1 - 0.67 gm  $CrO_3$  dissolved in 10 ml of tertamly alcohol to prepare tert amyl chromate and 3.04 gm Vanillin dissolved in 10 ml THF

Substrate/oxidant ratio 2:1 - 1.0 gm  $CrO_3$  dissolved in 10 ml of tert amyl alcohol to prepare tert amyl chromate and 3.04 gm of Vanillin dissolved in 10 ml THF

Substrate/oxidant ratio 1:1 - 2.0 gm  $CrO_3$  dissolved in 10 ml of tert amyl alcohol to prepare tert amyl chromate and 3.04 gm of Vanillin dissolved in 10 ml THF

Other solutions were prepared by standard methods.

#### 4.3 Procedure

Vanillin was found to be freely soluble in all the three selected solvents, namely, THF, dichloromethane and 1,4- dioxane. Oxidation of this substrate with TAC was performed in three sets, each for the three solvents. In each of these reactions sets, a solution of the substrate was prepared in THF, dichloromethane and 1,4-dioxane respectively. A solution of the oxidant, i.e., TAC was prepared separately in situ from Chromium trioxide and t-amylol, varying the substrate/oxidant molar ratio as 3:1, 2:1 and 1:1 for each set. The two solutions were then mixed together to prepare a homogenous reaction mixture. This mixture was then stirred continuously for a considerable time and any change in consistency was noted. The mixture was then heated in an LG MG 3937C 20- Litre, 2450 MHz 700-Watt Solo Microwave Oven (MW irradiation 160w, variable reaction time). Initial and final temperature of the reaction mixture was recorded to specify the

exothermic/endothermic nature of the reaction under investigation. The reactions were found to be exothermic, in general. The nine products formed in each of these cases were washed with acetone, dried and weighed, bottled and labeled as V-131, V-121, V-111, V-231, V-221, V-211, V-331, V-321, V-311 and was used for further analysis and characterization.

#### 4.4 Elemental analysis and preliminary physical characteristics

All the nine compounds obtained after the oxidation of Vanillin with TAC in different solvents were found to differ in basic physical properties like colour, magnetic properties and melting points. However, each of these products was insoluble in cold water but soluble in hot water. Magnetic

measurements show that the oxidation state of Cr in these compounds is +3. Percentage composition of C, H and O was determined using a Thermo Scientific Flash 2000 Organic Elemental Analyzer while the chromium content in these samples was estimated volumetrically using  $K_2S_2O_8$  (excess), 0.1 N  $K_2Cr_2O_7$  solution and 0.1 N Mohr's salt solution. Empirical formula of these complexes was then deduced. The recorded details have been summarized in table-1.

Sample Label	Solvent used	Substrate/oxidant ratio	Reaction time (in gm)	Yield (in gm)	Empirical formula of products
V-131	THF	3.04g/0.67g; 3:1	60	3.1	$CrC_9H_{14}O_7$
V-121	THF	3.04g/1.0g; 2:1	30	3.7	$Cr_2C_{13}H_{17}O_{12}$
V-111	THF	3.04g/2.0g; 1:1	20	6.0	$Cr_2C_{10}H_{11}O_{16}$
V-231	$CH_2Cl_2$	3.04g/0.67g; 3:1	75	1.75	$CrC_{11}H_{14}O_9$
V-221	$CH_2Cl_2$	3.04g/1.0g; 2:1	40	3.0	$Cr_2C_9H_{15}O_{11}$
V-211	$CH_2Cl_2$	3.04g/2.0g; 1:1	20	4.85	$Cr_2C_9H_{19}O_{11}$
V-331	1,4-dioxane	3.04g/0.67g; 3:1	40	3.0	$CrC_{12}H_{16}O_{10}$
V-321	1,4-dioxane	3.04g/1.0g; 2:1	30	5.3	$Cr_2C_{11}H_{14}O_9$
V-311	1,4-dioxane	3.04g/2.0g; 1:1	20	9.0	$Cr_2C_{12}H_{14}O_{13}$

#### 4.5 Interpretation of IR Spectra of the complexes

Fourier Transform Infrared (FTIR) spectroscopy is an important analytical used in the field of Chemical Science and technology and for advanced research. Analysis of the FT IR spectrum of the complexes obtained from vanillin in the present work has revealed some interesting characteristics about their composition and bonding. The results were recorded in the form of an FT IR spectrum for each sample. A typical IR spectrum obtained for the sample labeled V-111 has been shown in fig.-1.

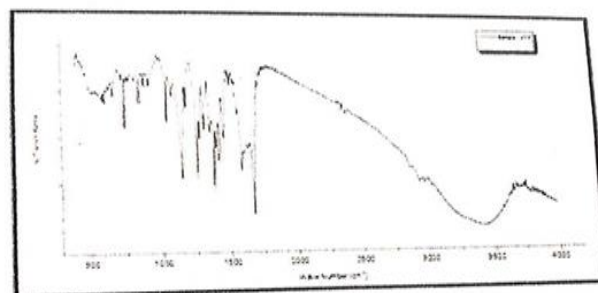


Fig. 1.A sample IR spectrum

Presence of Cr-O and Cr=O bonds in these complexes is indicated by the (Cr-O) stretching frequencies ranging from  $536-665\text{ cm}^{-1}$  and  $\nu$  (Cr=O) peaks near  $846, 847\text{ cm}^{-1}$ . The broad and strong absorption

bands appearing from 3200-3500  $\text{cm}^{-1}$  show the presence of H-Bonded (O-H) group, intensity and peak broadening varying greatly with variation in substrate/oxidant ratio. This indicates the difference in extent of coordination of the (O-H) oxygen atom with the Cr atom.

The lowest (O-H) stretching frequency being centered at 3310  $\text{cm}^{-1}$  in the compound labeled V-231 while the maximum value recorded in for V-331 and 3422  $\text{cm}^{-1}$ . A strong (C=O) carbonyl absorption is visible near 1675  $\text{cm}^{-1}$ . However, the absence of characteristic C-H stretching frequency for an aldehyde group suggests that the vanillin aldehyde group has been oxidized to a carboxylic group. This is further confirmed by the appearance of relevant peaks near 910  $\text{cm}^{-1}$  for (C-O) bending vibrations and one near 2969~2970  $\text{cm}^{-1}$  for (C-O) stretching vibrations of a carboxylic group. The characteristic peaks for aromatic (C-H) stretching, aromatic (C-C) and (C=C) in-ring stretching vibrations near 3021~3022  $\text{cm}^{-1}$ , 1422~1423  $\text{cm}^{-1}$  and 1586~1588  $\text{cm}^{-1}$  respectively indicates that the aromatic ring in Vanillin is intact and has not undergone any decomposition/cleavage during the course of oxidation. The presence of (C-O) stretching vibrations for the methoxy group near 1259  $\text{cm}^{-1}$  in the IR spectrum of these complexes is indicative of the oxygen atom of the ether group of Vanillin being uncoordinated. This is also supported by the weak alkyl (C-H) stretching vibrations appearing near 2924~ 2945  $\text{cm}^{-1}$  for each of these compounds. Other weak absorption bands for bending and rocking vibrations of (C-H) group were recorded near 744  $\text{cm}^{-1}$  and 1355~1357 respectively. A comparative spectral analysis for the nine oxidation products of vanillin (Chart-1 and Chart-2) shows considerable shift in the relative intensities and peak positions of these vibrational frequencies which may be attributed to a difference in the percentage composition of Cr metal and variable extent of its coordination with the ligand. The strong peaks at 1385  $\text{cm}^{-1}$  and 1633  $\text{cm}^{-1}$  might be due to the

impurities present in the KBr. A very weak peak near 2400  $\text{cm}^{-1}$  is characteristic of  $\text{CO}_2$  absorption.

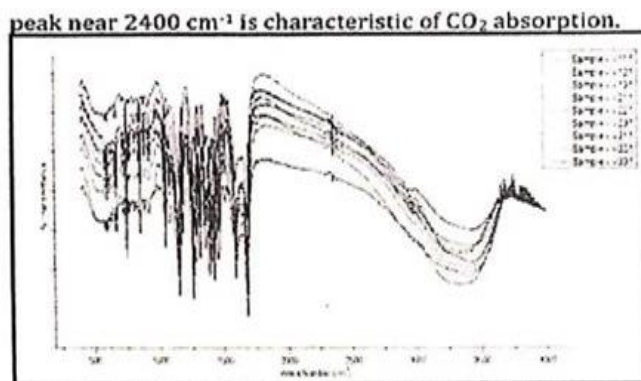


Chart-1: Comparison of relative intensities of the characteristic peaks in the IR spectrum of oxidation products of Vanillin (Plotted using Origin Pro 8.5 software)

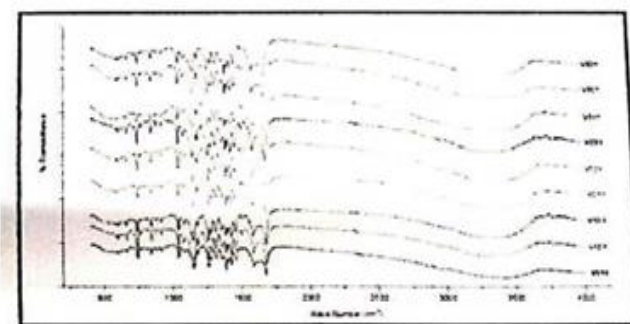


Chart-2: Comparison of relative positions of the characteristic's peaks in the IR spectrum of oxidation products of vanillin (Plotted using Origin Pro 8.5 software)

## V. CONCLUSION

Substrate/oxidant ratio has greatly affected the course of reaction, nature and characteristics of the compounds formed during the oxidation of 4-Hydroxy-3-methoxybenzaldehyde with TAC. It was also observed that the efficiency of these oxidation reactions depends upon the choice of solvent. The most efficiently synthesized product was the sample labeled V-311 in 1,4-dioxane. Most microwave assisted organic synthesis has been performed in domestic household microwave ovens using solvent-free technique, that is, carrying out the reactions on

solid support like - clays, alumina, silica, etc. Since this technology deals with high temperature and high-pressure conditions, safety will always be a main concern.

Besides, domestic household microwave ovens, which are readily available and inexpensive, reflux systems, pressurized systems and continuous flow system have also been used. On the basis of applied research in close collaboration with the standard national laboratories, the reaction summary, FT IR data and analysis presented in this research paper can contribute significantly to optimize the operating conditions of the various instruments used for synthesis and analysis, thereby increasing the efficiency of the microwave assisted oxidation of organic compounds and the quality of oxidation products obtained from them. The use of a graphical tool (Origin Pro 8.5) for data interpretation was found to be very helpful and this increases its scope in advanced and extensive research works. Furthermore, the obtained data may be implemented in the organic compounds databases and can be used for the simulation of oxidation processes.

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