Solvent Free Synthesis of Trialkylammonium Chromates (TAC) as Oxidative Promoter

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Abstract

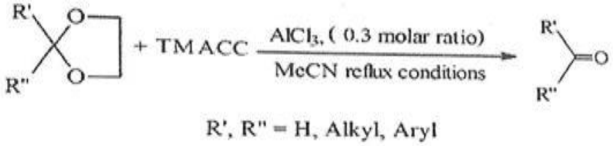
Trialkylammonium halochromates (R3NH[CrO3X]) (R = C2H5, C4H9, C3H7, CH3; X = F, Cl) are a beneficial addition to the oxidant series because of their affordability, availability, and effectiveness in oxidizing a wide range of organic compounds. A gentle and effective technique that uses trialkyl ammonium fluorochromates at room temperature and microwave radiation to oxidize diols to hydroxyl aldehydes offers a rapid, selective, and environmentally friendly approach, minimizing by-product formation and preserving sensitive functional groups during the oxidation process Traditional chromium-based oxidants frequently have problems such as poor selectivity, unfavorable reaction conditions, and hazardous byproducts that pose a threat to the environment. By exploring trialkylammonium chromates and halochromates, this study aims to overcome these challenges by identifying more effective and potentially greener alternatives for the oxidation of vanillin, while also examining how different halide ions influence the reaction outcome. The goal of the study is to comprehend how halide changes impact vanillin oxidation and aid in the creation of more potent and selective oxidants based on chromium. Our findings revealed that the nature of the halide significantly influenced the oxidation rate and product yield, with bromochromate showing higher reactivity. This study contributes to understanding the role of halide ligands in chromium (VI) oxidations and supports the development of more selective oxidants for aromatic compounds like vanillin.

Introduction

In recent years, numerous oxidative reagents have been introduced, achieving varying levels of success in terms of reactivity, selectivity, and environmental impact (Song, 2006; Goyal et al., 2022). Among these, chromium (VI)-based oxidants continue to entice concentration due to their excellent efficiency as well as selectivity in transforming a broad spectrum of organic compounds under mild conditions (Patnail et al., 2000; Zhang et al., 2024; Goyal et al., 2022). These reagents propose controlled oxidation with minimal by-product formation, making them valuable tools in synthetic organic chemistry. Significant advancements have also been realized through the use of quaternary ammonium salts in combination with chromium (VI) oxidants. Tetralkylammonium ions, including tetramethylammonium, tetraethylammonium, tetrapropylammonium, tetrahexylammonium, and tetrabutylammonium, have proven to enhance the solubility and reactivity of oxidizing agents in organic media. These cations are often paired with various chromium-based anions such as fluorochromate, chlorochromate, dichromate, and bromochromate to form phase-transfer complexes with improved performance characteristics. These systems not only facilitate effective oxidation but also allow for better control over reaction parameters. The synergy between tetralkylammonium ions and chromium-based oxidants has thus emerged as a promising approach for designing selective and efficient oxidative protocols (Patnail et al., 2000; Zhang et al., 2024; Goyal et al., 2022). To develop efficient oxidation methods, researchers have synthesized symmetric and asymmetric tetraalkylammonium ions with different alkyl chain lengths. In organic solvents, these ions act as oxidant carriers and promote reactions with organic substrates. They have been applied in solid-state processes, solvent-free systems, and microwave-assisted reactions. However, their effect on water structure remains unclear. Larger alkyl groups enhance water structuring, while smaller alkyl groups with greater exposure of charge on the onium ion led to the disruption of the water structure (Yoshihiro et al., 2008; Patel et al., 1972; Turner et al.,1992; Poos et al., 1953; Li et al., 2021).

Cr (VI) Oxidants and Alkyl Ammonium Ions as Carriers

In laboratories, water-soluble potassium or sodium dichromates are frequently employed to oxidize organic substrates, particularly when strong acids are present. Pyridine was first used as an organic phase transfer candidate by researchers at the Sarett Research group. It forms a salt with the Lewis acid CrO_3 to aid in the oxidation of steroidal alcohols in solvents (organic solvent) (Bernstein et al., 1960; Chawala et al., 1974; Gilbert et al., 1976). Subsequently, other researchers used the reagent without looking into its structure (oxidant). Corey revisited Sarett's reagent and identified it as pyridinium dichromate in his groundbreaking work to establish pyridinium chlorochromate as a versatile oxidant (Bernstein et al., 1960; Chawala et al., 1974; Gilbert et al., 1976; Corey; 1975; Corey et al., 1979; Heravi et al., 2016).Following that, a range of heterocyclic ammonium ion-based Cr(VI) oxidants were created, and their capacity to oxidize different substrates was investigated (Patel et al., 2007; Liang et al., 2021). These oxidants were thoroughly reviewed in a publication (Sadeghy et al., 2005). The kinetics of methionine (Met) oxidation by Triethylammonium Chromate Complex (TEACC) in Dimethyl sulfoxide (DMSO), which yields the equivalent sulfoxide, were recently investigated by Patel et al. in 2007. It was discovered that the reaction is catalyzed by hydrogen ions and is first-order with regard to both Met and TEACC.

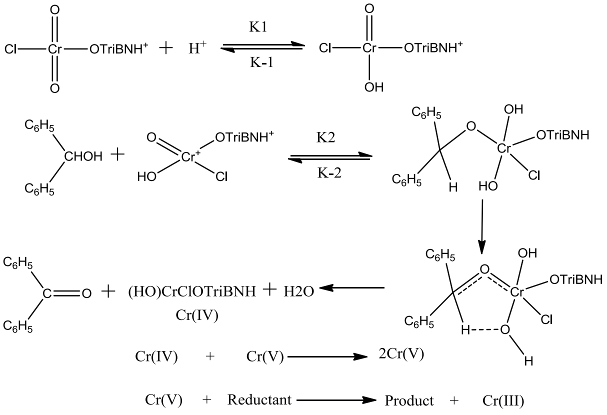


Scheme1(Guo et al., 2025)

In a different study, Chouhan et al. used benzyltriethylammoniumchlorochromate (BTEACC) in DMSO to examine the conversion of aliphatic aldehydes to carboxylic acids and the oxidation of methanoic and ethanedioacid acids to yield Carbondioxide. Additionally, primary alcohols were oxidized to aldehydes using BTEACC. Other types of Cr (VI) oxidants include tetramethylammonium chlorochromate (TMACC:$(CH_3)_4N[CrO_3]Cl$) and tetramethylammoniumfluorochromate (TMAFC: $(CH_3)_4N[CrO_3]F$).

The corresponding quaternary ammonium compounds are reacted with CrO_3 in acetonitrile in an equimolar (1:1) ratio to create these reagents. Trimethylsilyl and tetrahydropyranyl ethers, ethylene acetals and ketals, and other molecules have been oxidatively deprotected by TMACC, which transforms them into their respective carbonyl compounds (Scheme-1). Additionally, it has been applied to the oxidation of aliphatic and aromatic thiols to their respective disulfides. The crystal and molecular structures of TMAFC were investigated using X-ray diffraction at 130 K. The data show a difference between the Cr–O and Cr–F bond lengths, similar to TEACC, which is probably due to a CH.F hydrogen bond between the methyl hydrogen (cation) and fluoride (anion).

It has been demonstrated that TMAFC effectively oxidizes n-butyl, isopropyl, and benzyl alcohols into the corresponding aldehydes. When p-toluenesulfonic acid was present, the kinetics of this oxidation process were examined. The findings showed that, in terms of the alcohols, the reaction proceeds according to Michaelis-Menten kinetics. This implies that the oxidizing agent and the alcohol will create a quasi-equilibrium combination. The C-H bond at the carbon bonded to the hydroxyl group cleaves, according to the kinetic isotope effect seen for benzyl alcohol. As seen in Scheme-2, a hydride transfer mechanism was put up to explain the oxidation process. The oxidation of several aliphatic, aromatic, and allylic thiols to their corresponding disulfides was also shown by Imanieh et al. In these reactions, TMAFC is reduced by two electrons; no sulphones or sulphonic acids are formed.



Scheme2 (Sahu et al., 2017)

Trialkylammonium Chromates

In the class of adaptable oxidants, trialkylammoniumhalochromates (R_3NH [CrO3X]) (Where

R = Me, Et, Pr, and Bt and X = F, Cl) are a noteworthy addition. These chemicals can effectively oxidize a variety of organic substrates and are affordable and easily accessible. A gentle and effective method for oxidizing diols to their corresponding hydroxy aldehydes using trialkylammoniumfluorochromates (R3NH [CrO3X]) (R = Me/CH3, Et/C2H5, Pr/C3H7, and Bt/C4H9) under both normal and microwave irradiation was reported by researchers (Hajipour et al., 2005; Santaniello et al., 1983; Ghammamy et al., 2005; 2007; Pourali et al., 2006).

Primary and secondary alcohols in dichloromethane have been oxidized using tribylammoniumchlorochromate (TriBACC), producing high yields of the related aldehydes and ketones. Tributylamine, hydrochloric acid, and CrO3 are reacted in a 1:2:2 molar ratio to create the reagent. Mansoor et al. have investigated the oxidation kinetics of benzhydrols to benzophenones using TriBACC. In terms of TriBACC, benzhydrol, and H+ concentrations, the reaction proceeded according to first-order kinetics. For substituted benzhydrols, the reported reactivity sequence was p-OCH3 > p-CH3 > p-H >> p-Cl > p-NO2. Scheme 2 and 4 shows the suggested hydride transfer mechanism for the reaction. Tripropylammoniumfluorochromate (TriPAFC) has been used to convert anthracene and phenanthrene into anthraquinone and phenanthraquinone, respectively, and to oxidize alcohols to their respective aldehydes or ketones. Both in solution and when exposed to microwave radiation, tripropylammoniumchlorochromate (TriPACC) has been used to oxidatively couple thiols into disulfides (Scheme 3). Furthermore, TriPAFC and TriPACC have both been used to oxidize thiols to their corresponding disulfides when adsorbed on alumina in solution.

Primary alcohols, anthracene, naphthacene, and carbohydrates may all be efficiently oxidized by triethylammoniumfluorochromate (Et3NHCrO3F) into their respective oxo derivatives in dichloromethane with good yields (Scheme 4). The transformationtriethylammoniumfluorochromate or triethylammoniumchlorochromate supported on silica gel is used to convert a number of aliphatic and aromatic thiols into disulfides was also reported by Ghammamy et al. 2005: 2007; 2008.CrO3, trimethylamine, and 40% aqueous HF were combined in a molar ratio of 1:1:2 to create trimethylammoniumfluorochromate (TriMAFC). The appropriate carbonyl compounds in dichloromethane were then obtained by applying it to the oxidation of alcohols.

$$\mathrm{RSH} \xrightarrow[\text{A}]{\text{TriPACC}} \mathrm{RSSR}$$

orB

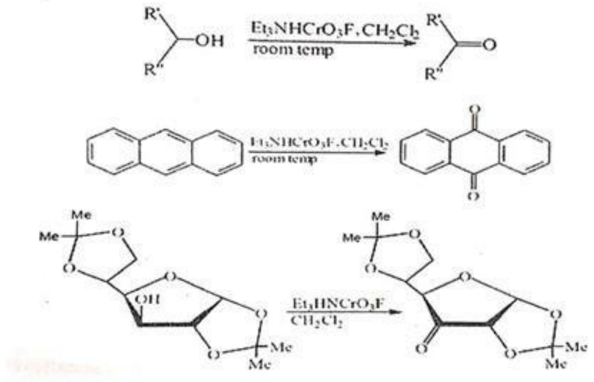
A: CH_2Cl_2rt

B: CH2Cl2.rtmicrowave

Scheme3

Dialkylammonium Chromates

In an aqueous acetic acid solution, primary and secondary alcohols were oxidized into their corresponding carbonyl compounds using diethylammoniumchlorochromate (DEACC), a form of dialkylammonium chromate. About both DEACC and H^+, the reaction was shown to follow first-order kinetics, displaying a Michaelis-Menten type pattern. Dimethylammonium chlorochromate (DMACC) showed notable effectiveness for alcohol oxidation, benzoin transformation, and the oxidative breaking of C=N bonds to regenerate carbonyl compounds under non-aqueous circumstances when chemisorbed on alumina and silica.N-methylbenzylammoniumfluorochromate (MBAFC) and N-ethylbenzylammoniumfluorochromate (EBAFC) were created by Sayyed-Alangi and his group to selectively oxidize alcohols to carbonyl compounds. When these chemicals were adsorbed on silica gel, their activity was greatly increased. These oxidants also exhibited exceptional chemo-selectivity because they did not react with functional groups like thiols, sulfides, or phenols, which made them more useful for creating highly functionalized compounds. By handling Chromium trioxide with aqueous HF and either N-methylbenzylamine or N-ethylbenzylamine in a 1:1.5:1 molar ratio, MBAFC and EBAFC were produced.



Scheme 4 (Katre. 2020; Sahu et al., 2017)

Alkylammonium chromates

Using methylammonium chlorochromate (MCC) adhering to silica gel (SiO2), carbonyl compounds were successfully regenerated from derivatives that contain nitrogen (such as oximes, p-nitrophenylhydrazones, 4-phenylsemicarbazones, and semicarbazones). Additionally, this chemical proved successful in converting silica's hydroxyl groups into their matching carbonyl compounds. The kinetics of ammonium chlorochromate (ACC)-induced phenol oxidation to quinones in an aqueous acetic acid media were investigated by Patwari et al. Their results showed that hydrogen ions catalyzed the process, which had first-order kinetics for both phenol and ACC. As the solvent's dielectric constant rose, the rate of oxidation slowed, indicating ion-dipole interactions during the oxidation process. The combination of protonated ACC with chloride ions produced a reactive species, which was responsible for the decrease in oxidation rate observed with an increase in KCl concentration. Using Li and colleagues supported ACC on montmorillonite K10 in dichloromethane also reported oxidizing hydrobenzoins to their corresponding benzyl compounds.

Results and Discussion

Edgar Hugo Emil Reinsch (1809–1884) was the first to isolate 1,2-Dihydroxybenzene, usually referred to as catechol, in 1839. Catechin, a solid preparation made from catechu, the boiled or condensed juice of Mimosa catechu (now Acacia catechu L.f.), is where he got it by distilling it. Reinsch called the white efflorescence that resulted from heating catechin above its breakdown point "Brenz-Katechusaure," or "burned catechu acid." It was a byproduct of catechin's flavanols' heat degradation. Both Wackenroder and Zwenger independently rediscovered catechol in 1841, and the substance was dubbed pyrocatechin in the Philosophical Magazine. August Kekulé verified catechol's status as a benzene diol in 1867 after Erdmann recognized it as a benzene derivative with two oxygen atoms by 1852. In 1868, the chemical was identified as pyrocatechol. The name "catechol," which was proposed by the Journal of the Chemical Society in 1879, became the commonly used term in the years that followed.

4-Hydroxy-3-methoxybenzaldehyde (Vanillin) oxidation using TAC (Ditertiary amyl chromate)

TAC was used to oxidize 4-hydroxy-3-methoxybenzaldehyde (vanillin) and create the Cr (III) complexes with the help of microwave heating. This method, in comparison to conventional heating techniques, demonstrated improved reaction efficiency, higher yield, better specificity, reduced pollution, and energy efficiency. The products were characterized. It had been observed that the properties of the products are significantly influenced by the ratio of substrate to oxidant and the solvent used in each reaction. Vanillin (C8H8O3), primarily derived from vanilla beans, has been extensively studied, though there has been limited exploration of its reaction with TAC. TAC is a versatile reagent, known for oxidizing organic compounds and forming metal complexes. A relatively novel technique, has gained popularity among chemists due to its environmental advantages, offering a more energy-efficient and green approach to chemical synthesis. Despite its growing use, further improvements in microwave technology and instrumentation for scientific research remain necessary. This study focuses on the microwave-assisted synthesis of chromium complexes from vanillin with TAC, using various solvents such as tetrahydrofuran (THF), dichloromethane, and 1,4-dioxane, and presents a comparative analysis of their characterization through FTIR spectroscopy (Tomar et al., .2006;Tomar and Kumar, 2006; 2007; 2009; 1320;Ghammamy and Baghy., 2008; 2008).

Preparation of solutions: Preparation of TAC

TAC was made in situ in this investigation by dissolving a certain quantity of chromium (VI) oxide in 10 milliliters of tert-amyl alcohol, which served as the solvent.

Preparation of reaction mixture

To prepare the reactions with different substrate/oxidant ratios, dissolve 0.67 g of CrO_3 in 10 mL of tert-amyl alcohol for a 3:1 ratio, 1.0 g of CrO_3 in 10 mL of tert-amyl alcohol for a 2:1 ratio, and 2.0 g of CrO_3 in 10 mL of tert-amyl alcohol for a 1:1 ratio to form tert-amyl chromate.

Methods

THF, dichloromethane, and 1,4-dioxane were the three solvents that vanillin was soluble in. Three different experiments were carried out, each utilizing one of these solvents to oxidize vanillin with TAC. Vanillin was dissolved in 1,4-dioxane, dichloromethane, or THF in each experiment. Chromium trioxide and t-amyl alcohol were combined to create an in situ solution of the oxidant, TAC, with different substrate-to-oxidant molar ratios of 1:1, 3:1, and 2:1 for distinct situations. To get a consistent reaction mixture, the two solutions were combined. After that, this mixture was constantly mixed, and any consistency changes were noted. After that, the chemical (reaction) was heated in an LG MG 3937C 20-Litre, 2450 MHz 700-Watt Solo microwave oven with 160W of microwave irradiation, and the reaction time was varied. To ascertain whether the reaction was exothermic or endothermic, the initial and final temperatures were noted. The reactions were generally exothermic. Each reaction produced nine products, which were then designated V-131, V-121, V-111, V-231, V-221, V-211, v-331, V-331, and V-321 after being cleaned with acetone, dried, and weighed. They were then ready for additional examination and characterization.

Elemental analysis and preliminary physical characteristics

The nine compounds that were produced by oxidizing vanillin with TAC in different solvents showed variations in their fundamental physical characteristics, including color, melting temperatures, and magnetic behavior. All of the products were insoluble in cold water but soluble in hot water, notwithstanding these variations. These compounds' chromium was shown to be in the +3 oxidation state by magnetic examination. A Thermo Scientific Flash 2000 Organic Elemental Analyzer was used to measure the percentage compositions of carbon, hydrogen, and oxygen. Excess K2S2O8, 0.1 N K2Cr2O7 solution, and 0.1 N Mohr's salt solution were used to estimate the chromium concentration volumetrically. The empirical formulas for the complexes were inferred from these analyses. Table 1 contains the condensed data.

Table1. *Elemental analysis*

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	Cr2C13H17O12
V-111	THF	3.04g/2.0g;1:1	20	6.0	Cr2C10H11O16
V-231	CH_2Cl_2	3.04g/0.67g;3:1	75	1.75	CrC11H14O9
V-221	CH_2Cl_2	3.04g/1.0g;2:1	40	3.0	Cr2C9H15O11
V-211	CH_2Cl_2	3.04g/2.0g;1:1	20	4.85	Cr2C9H19O11
V-331	1,4-dioxane	3.04g/0.67g;3:1	40	3.0	CrC12H16O10
V-321	1,4-dioxane	3.04g/1.0g;2:1	30	5.3	Cr2C11H14O9
V-311	1,4-dioxane	3.04g/2.0g;1:1	20	9.0	Cr2C12H14O13

Interpretation of the Complexes' IR Spectra

A vital analytical method that is frequently employed in advanced research in the chemical sciences is Fourier Transform Infrared (FTIR) spectroscopy. The complexes created by oxidizing vanillin in this study had FTIR spectra that provided intriguing information about their bonding and structural makeup. Each complex's spectral data was captured, and Fig. 1 displays a sample FTIR spectrum for the chemical designated V-111. The presence of Cr-O and Cr=O bonds in the complexes is confirmed by the stretching frequencies linked to the Cr-O bond, which range from 536 to 665 cm^{-1}, and the Cr=O stretching peaks, which are located at roughly 846-847 cm-1 (Socrates 2004; Nakamoto 2009). A wide and strong absorption band is also observed (Socrates 2004). The substrate-to-oxidant ratio significantly affected the band's width and intensity, indicating variations in the (O-H) oxygen atom's coordination with chromium. Compound V-231 had the lowest (O-H) stretching frequency, measuring 3310 cm-1, whereas compound V-331 had the highest, measuring 3422 cm-1. Around 1675 cm-1, a noticeable carbonyl (C=O) absorption was seen. The lack of the aldehyde C-H stretching frequency indicates that vanillin's aldehyde group has been converted to a carboxylic group. This is corroborated by peaks at about 2970 cm-1 for (C-O) stretching and 910 cm-1 for (C-O) bending vibrations, which are characteristic of carboxylic acids. Vanillin's aromatic structure is unaltered by oxidation, as demonstrated by the aromatic C-H stretching, C-C, and C=C in-ring vibrations at 3022 cm-1, 1588 cm-1, and 1423 cm-1 (Socrates 2004; Pretsch et al., 2013; Nakamoto 2009). Weak alkyl C-H stretching bands near 2945 cm-1 in all the complexes support the methoxy group's (C-O) stretching vibrations near 1259 cm-1, which show that the ether group's oxygen is not coordinated with the chromium atom. Near 744 cm-1 and 1357 cm-1, additional weak absorption bands for (C-H) bending and rocking vibrations were detected (Socrates 2004; Nakamoto 2009). Variations in the chromium concentration and the degree of coordination with the ligand may be the cause of the discernible shifts in the intensities and locations of various vibrational peaks found in the FTIR spectra of the nine vanillin oxidation products (Figure 2). A very small signal close to 2400 cm-1 indicates CO2 absorption, while the strong peaks at 1385 cm-1 and 1633 cm-1 are probably caused by KBr impurities.

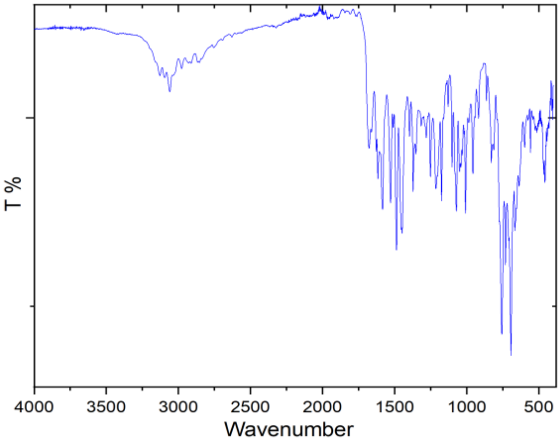


Figure 1. A sample (V-111) IR spectrum

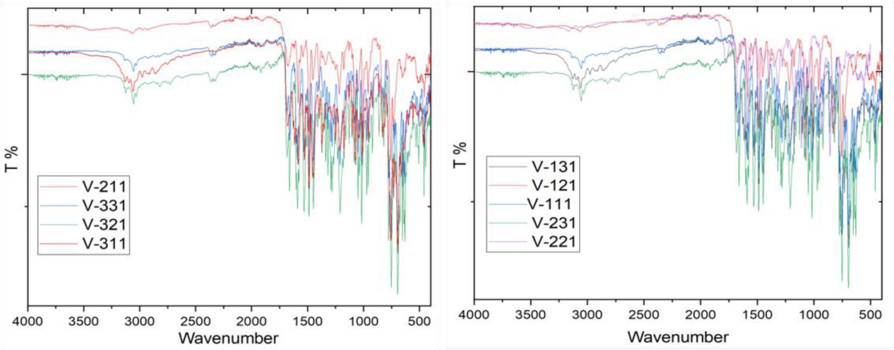


Figure 2 FTIR of nine vanillin oxidation products

Conclusion

The development, type, and properties of the molecules generated during the oxidation of 4-hydroxy-3-methoxybenzaldehyde with TAC are significantly influenced by the ratio of substrate to oxidant. Furthermore, the solvent selection affects how effective these oxidation reactions are. The most efficient product among the samples was V-311, which was produced in 1,4-dioxane. The majority of microwave-assisted organic synthesis techniques have been carried out in home microwave ovens, usually using a solvent-free method and conducting reactions on solid supports like silica, alumina, or clays. Safety is the top priority because these techniques involve high pressure and temperature conditions. Other devices such as continuous flow systems, pressured systems, and reflux systems have also been utilized in addition to home microwave ovens. In order to improve the effectiveness of oxidation with microwave assistance reactions and the quality of the products produced, the reaction data, FTIR analysis, and conclusions presented in this paper can greatly aid in improving the operating conditions of different synthesis and analysis instruments through cooperative research with national laboratories. Origin Pro 8.5, a graphical tool, was particularly useful for data analysis, expanding its applicability to more complex and thorough research. Additionally, the information can be added to databases of organic compounds in order to simulate oxidation processes.

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