

Kinetics and oxidation of Co (III) complexes of α -Thio acids by 2, 2'- bipyridinium bromo chromate in the presence of miceller medium

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ABSTRACT: Kinetics of oxidation Cobalt (III) complexes of α -Thio acids by 2, 2'-bipyridinium bromo chromate (BPBC) in the presence of miceller medium at 55^oC in 50% acetic acid – 50 % water. The reaction the rate of oxidation shows pseudo first order kinetics. BPBC oxidizes Cobalt (III) bound and unbound α -Thio acids. It rules out the synchronous C-C bond fission and electron transfer to Cobalt (III) Centre. The reaction was followed by observing the decrease in the absorbance at 365 nm for the Cr (VI) present in the reagent in a UV – Visible spectrophotometer. Product and stoichiometric analysis were carried out for the oxidation of complexes and free ligands. A mechanism involving one electron transfer for the ligand was proposed.

KEYWORDS: α -Thio acids, 2, 2'-bipyridinium Bromochromate, Sodium lauryl sulphate, Cetyl trimethyl ammonium bromide, TRITON-X 100.

I. INTRODUCTION

Chromium compounds have been widely used in aqueous and non-aqueous medium for [1]²⁻⁵ the oxidation of a variety of organic compounds, chromium compounds especially Cr (VI) reagents have been proved to be versatile reagents capable of oxidising almost all the oxidisable organic functional groups [1]^{6, 8}. Generally Cr (VI) oxidation reactions have been performed in aqueous acidic conditions, the source of Cr (VI) being chromium trioxide and sodium or potassium dichromate. Aqueous sulphuric acid and acetic acid are the most frequently employed acids, acetone, benzene, methylene chloride or ether are often used as co-solvents in the case of water insoluble organic compounds. Narayanan and Balasubramaniyan [9] have found PBC as an efficient oxidant for alcohols as well as a brominating agent for aromatic compounds. Banerji *et al* [10] studied the kinetics of oxidation of thioglycollic acid, thiolactic acid by BPBC. The reaction is first order with respect to [BPBC] and Michaelis-Menten types of kinetics were observed with respect to all the thioacids. The rate was not affected by the addition of acrylonitrile indicates the absence of the free radical mechanism. From the results, a suitable mechanism was proposed as follows. 2, 2'-bipyridinium bromo chromate (BPBC) has been used as a mild, efficient and selective oxidising reagent in synthetic organic chemistry. 2, 2'-Bipyridinium chlorochromate (BPCC), has been used as a mild and selective oxidant in synthetic organic chemistry [11]. There are only a few reports about the kinetics and mechanism of oxidation by BPCC, available in the literature [12-15], while the kinetics of oxidation of organic sulphides by BPCC has not been investigated.

II. EXPERIMENTAL SECTION

2.1. PREPARATION OF BPBC

2, 2'-Bipyridine (A.R. Qualigens, India), chromium trioxide from (SD Fine chemicals, India 95%) Hydrobromic acid (47%, Merck, India) Preparation of BPBC to 16.75 ml of 6 M hydrobromic acid (0.11mole) is added 10.0 g. (0.1 mole) of chromium trioxide rapidly while stirring. After dissolution of the chromium trioxide is complete, 15.6 g. (0.1 mole)

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of 2, 2'-bipyridine is added in portions while stirring vigorously. A yellow slurry results which is stirred for 1 hour at room temperature. The slurry is then collected on a sintered glass funnel and washed with two 15 ml portions of cold distilled water. The resulting solid yellow filter cake is dried for 3 hours in vacuum at room temperature. The resulting product is 2, 2'-bipyridinium bromochromate, its purity was checked by an iodometric method and is obtained in a typical yield of 26.8 g. which is 92% of the theoretical yield. Thiolactic, Thiomalic and Thioglycolic acids from (SD Fine chemicals. India 95%)

2.2. COBALT (III) COMPLEXES OF α -THIO ACIDS

The cobalt (III) complexes of alpha-Thio acids were prepared as their perchlorates using the method of Fan and Gould [16]. The surfactants used in the present work are sodium lauryl sulphate [17] (NaLS) and cetyl trimethylammonium bromide [18] (CTAB). The surfactants are purified by adopting earlier procedure[19,20,21] Kinetic measurements of the oxidation of cobalt (III) complexes of alpha-Thio acids and unbound ligands were carried out under pseudo first order conditions in 50% acetic acid 50% water at $55 \pm 2^\circ\text{C}$ by following the concentration of BPCC at 365 nm with the help of an UV visible spectrophotometer. The total volume of reaction mixture in the spectrophotometric cell was kept as 2.5 ml in each kinetic run all the solutions for the experiments were maintained at the specified temperature in an electrically operated thermo stated bath. Perchloric acid was used as the source of H^+ ions and ionic strength was maintained by NaClO_4 .

III.KINETIC MEASUREMENTS

All kinetic measurements were carried out by Spectrophotometer. The progress of the reaction was followed at 365 nm by monitoring the changes in absorbance of remaining Cr (VI) and 502nm by monitoring the changes in absorbance of remaining Co (III). The required α -Thio acid, HClO_4 and BPBC were premixed in a reaction vessel, thermo stated in an oil bath, and BPBC solution was then added prior to the absorbance measurements. Under pseudo-first-order conditions of α -Thio acid, the plots of $\log A$ versus time (A is absorbance intensity) were linear up to 85% completion of the reaction with an average of linear regression coefficients, $r \geq 0.998$. The rate of oxidation of Thio-Glycolic depends on BPBC concentration, the specific rate calculated remains constant (Table 2) and Graph of logarithm of BPBC concentration versus time (Fig. 1) are linear. From the slope of these graphs, the specific rate calculated agrees with those obtained from integrated rate equation suggesting pseudo first order dependence on BPBC concentration.

IV.PRODUCT ANALYSIS

Product analysis was carried out under kinetic conditions *i.e.*, with excess of the reductant over BPBC. In a typical experiment, Thio lactic acid (0.10 mol), perchloric acid (0.10mol) and BPBC (0.01 mol) were dissolved in acetic acid – water mixture (50%- 50%) and the solution was allows to stand in the dark for about 24 h to ensure completion of the reaction. The residue was treated with an excess (200 mL) of a saturated solution of 2, 4-dinitro phenyl hydrazine in 1mol HCl and kept overnight in a refrigerator. The precipitated 2, 4-dinitro phenyl hydrazone (DNP) was filtered off, dried and recrystallised from ethanol. The product was identical with melting point and mixed melting point to an authentic sample of the DNP of acetaldehyde.

V.STOICHIOMETRY

Stoichiometric studies for the oxidation of pentaammine cobalt (III) complexes of alpha-thio acids and free ligands by BPBC were carried out with the oxidant in excess. The $[\text{H}^+]$ and ionic strength were maintained as such in the corresponding rate measurements. After ten half-lives when the reaction was nearing 80% completion the concentration of unreacted BPBC was determined spectrophotometrically.

VI.RESULT AND DISCUSSION

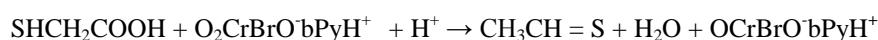
The kinetics and oxidation of alpha thio acids by 2,2'-bipyridinium bromo chromate in micellar medium at $55 \pm 0.2^\circ\text{C}$ under pseudo first order condition and their bound Co^{III} complexes by BPBC in presence of perchloric acid of anionic

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and cationic micelles at $55 \pm 0.2^\circ\text{C}$. An explanation for the higher reactivity of Thio lactic acid involving C-H cleavage was given based on the higher acidity of C-H proton. In the present investigation, the absence of such an observation leads to C-C cleavage. The product analysis confirmed the formation of aldehydes due to C-C cleavage and not keto acids as a product. Further the absence of keto complexes in the product and formation of aldehydes as the only organic product confirm the C-C cleavage. The reduction of Co (III) to Co (II) to an extent of 98% showed an induced electron transfer from the ligand to the metal due to decarboxylation and synchronous cleavage of the C-C bond in the complexes. BPBC undergoes a one electron change and the overall reaction between BPBC and the complexes could be written as:



The rate equation for this reaction could be deduced as Rate = k [alpha-Thio acid] [BPBC]

The reaction proposes that BPBC oxidizes OH center of the cobalt (III) bound α - Thio acids at a rate comparable to that of the unbound ligand and there is 99 % reduction at the cobalt (III) center. The kinetic data for the oxidation of the pentaamminecobalt (III) complexes by BPBC in presence of Perchloric acid catalyst and maintained temperature $55 \pm 0.2^\circ\text{C}$.



The rate equation for the oxidation could be deduced as Rate = k [Co^{III} - Complex] [BPBC]

6.1 Effect of acidity

The reaction is catalyzed by hydrogen ions. The acid-catalysis may well be attributed to a protonation of BPBC to give a stronger oxidant and electrophile.



Table.1 shows Stoichiometric Data for BPBC oxidation of Co (III) bound and unbound α -Thio acids in presence of Sodium Lauryl Sulphate (NaLS) at $55 \pm 0.2^\circ\text{C}$.Stoichiometric analysis of the oxidation reaction nearly 98 %completion.

Table 1

[HClO₄] = 1.00×10^{-1} mol dm⁻³ [NaLS] = 1.00×10^{-2} mol dm⁻³ Temperature = $55 \pm 0.2^\circ\text{C}$

10^2 [Compound] mol dm ⁻³	10^2 [BPBC] _{Initial} mol dm ⁻³	10^2 [BPBC] _{Final} mol dm ⁻³	$\Delta 10^2$ [BPBC] mol dm ⁻³	[Compound] : Δ [BPBC]
Thio Lactic acid				
1.0	10.0	9.22	0.78	1.00 : 0.78
2.0	10.0	8.52	1.48	1.00 : 0.76
3.0	20.0	17.69	2.31	1.00 : 0.77
Thio Glycolic acid				
1.0	10.0	9.26	0.74	1.00 : 0.74
2.0	10.0	8.50	1.50	1.00 : 0.75
3.0	20.0	17.81	2.19	1.00 : 0.73
Thio Malic acid				
1.0	10.0	9.28	0.72	1.00:0.74
2.0	10.0	8.53	1.47	1.00:0.73
3.0	20.0	17.74	2.26	1.00:0.75
Co^{III} Thio Lactato				
1.0	10.0	9.64	0.36	1.00 : 0.36
2.0	10.0	9.30	0.70	1.00 : 0.35
3.0	20.0	18.98	1.02	1.00 : 0.34
Co^{III} Thio Glycolato				
1.0	10.0	9.66	0.34	1.00 : 0.34
2.0	10.0	9.24	0.76	1.00 : 0.33
3.0	20.0	19.04	0.96	1.00 : 0.32
Co^{III} Thio Malato				

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1.0	10.0	9.65	0.35	1.00:0.35
2.0	10.0	9.26	0.74	1.00:0.34
3.0	20.0	19.01	0.99	1.00:0.33

Table 2 shows the kinetic rate data on the oxidation of alpha thioglycolic acid by 2, 2'-bipyridinium bromochromate in the presence of micelle CTAB.

Table 2
[BPBC] = 10^{-3} mol dm⁻³ [HClO₄] = 0.1 mol dm⁻³ Temperature = $55 \pm 0.2^\circ\text{C}$
 α -Thio Glycolic Acid = 1.00×10^{-2} mol dm⁻³ [CTAB] = 1.00×10^{-2} mol dm⁻³

Time(s)	Log(Absorbance)	$10^4 k_1$ (s ⁻¹)
0	-0.18842	0.00
308	-0.2321	3.266
606	-0.27084	3.132
906	-0.31515	3.221
1202	-0.36151	3.316
1510	-0.40782	3.346
1807	-0.46092	3.473
2105	-0.50307	3.442
2403	-0.55596	3.522
2709	-0.60033	3.502
3008	-0.64782	3.517

Figure.1 shows the oxidation of thioglycolic acid by BPBC in the presence of surfactant CTAB at 328 K. The first order plots of log absorbance versus time in seconds were linear and regression is 0.998 and average rate constant k_1 is $3.374 \times 10^{-4} \text{ S}^{-1}$ from the Table 2

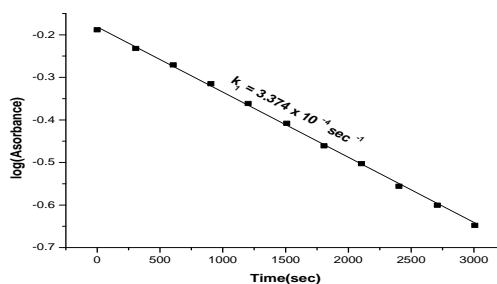


Fig.1

6.2. Effect of Temperature of the alpha- Thio acid

The reaction has been carried out at five different temperatures keeping [BPBC] = 10^{-3} mol dm⁻³ Thio Acid = 1.00×10^{-2} mol dm⁻³. Absorbance decrease the while reaction time of BPBC with alpha thio acid. The rate constants were calculated by the integrated rate equation. The graph of logarithm of absorbance versus time was linear and the rate constants calculated from the slope of the graph. Temperature increases rate also increased. The rate of the reaction as followed pseudo first order kinetics. The rate of cationic micelles much more the anionic micelles and neutral micelles.

6.3. Effect of Varying Substrate Concentration

The substrates are the concentration of the Thio Lactic acid, Thio Malic acid, Thio Glycolic acid (Table-3) in the presence of micelles were varied in the concentration range of 0.5×10^{-2} to 2.5×10^{-2} mol dm⁻³ at 328 K and keeping all other reactant concentrations as constant and the rates were measured. The substrates are the concentration of Co (III) lactate and Co (III) glycolato (Table-3) in the presence of micelles were varied in the range of 1.0×10^{-2} to 3.0×10^{-2} mol dm⁻³ at 328 K and keeping all other reactant concentrations as constant and the rates were measured. The free Thio

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acids and their Co (III) complexes of rate constants were calculated by the integrated rate equation. The graph of logarithms was concentration versus time linear and the rate constants calculated from the slope of the graph agreed with the experimental value. Which shows a first order dependence plot on α -Thio acids and their complexes. The rate of oxidation increased progressively with increasing the concentration of α -Thio acids in the presence of CTAB (Fig 2) and their complexes in presence of TRITON (Fig 3). The plot of $\log k_1$ versus $\log [\alpha\text{-Thio acids}]$ and Co (III) complexes of Thio acids gave the slope of ≥ 0.999 .

6.4. Effect of Varying Perchloric acid Concentration

The source H^+ in the reaction medium has been used by Perchloric acid. The concentration of perchloric acid was varied in the range 0.06 to 0.14 mol dm^{-3} keeping all other reactant concentration as constant at 328 K as well as Co (III) complexes and the rates were measured. The acid catalyzed by BPBC oxidation is confirmed by an increases in the rate with the addition of Perchloric acid. The plot of $\log k_1$ versus $\log [H^+]$ is a straight line with a slope of ≥ 0.998 . The varying H^+ concentration oxidised by BPBC with micelles the rate of the reaction CTAB higher than the TRITON, NaLS respectively. Table-3 shows the kinetic data for the oxidation of free ligand α -thio acids and Co (III) complexes of α -Thio acids by BPBC in the presence of CTAB with varying substrate concentrations and keeping all other reactant concentrations as constants and rates were measured at 328 K .

Table -3.
[BPBC] = $10^{-3} \text{ mol dm}^{-3}$ $[HClO_4] = 0.1 \text{ mol dm}^{-3}$ [Micelles] = $1.00 \times 10^{-4} \text{ mol dm}^{-3}$

$10^2[\alpha\text{-Thio acid}]$ mol dm^{-3}	$10^4 k_1 (\text{s}^{-1})$								
	NaLS	TRITON	CTAB	NaLS	TRITON	CTAB	NaLS	TRITON	CTAB
	Thio Glycolic acid			Thio Malic acid			Thio Lactic acid		
0.5	1.231	1.372	1.688	1.479	1.663	1.939	1.801	1.956	2.157
1.0	2.463	2.743	3.373	2.978	3.273	3.870	3.604	3.914	4.312
1.5	3.692	4.118	5.064	4.477	4.965	5.868	5.408	5.876	6.471
2.0	4.924	5.494	6.744	6.011	6.501	7.792	7.212	7.830	8.632
2.5	6.160	6.865	8.435	7.550	8.336	9.714	9.023	9.793	10.795

Figure.3 shows the plot of \log of α -Thio acids versus $\log k_1$ for the oxidation of α -Thio Lactic acid, α -Thio Malic acid and α -Thio Glycolic acid by BPBC in the presence of CTAB at 328 K . The rate of oxidation increased progressively with increasing the concentration of α -Thio acids. The slope is 0.998 measured

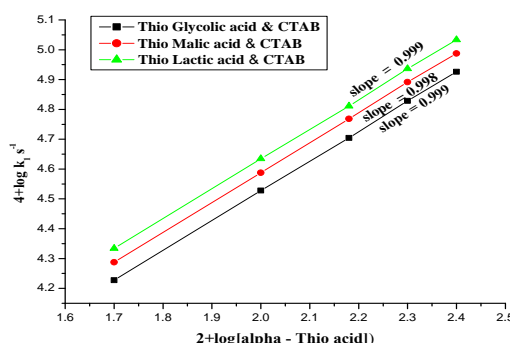


Fig 2

Table 4 shows the kinetic data for the oxidation of bound α -thio acids and Co(III) complexes of α -Thio acids by BPBC in the presence of TRITON with varying substrate concentrations and keeping all other reactant concentrations as constants and rates were measured at 328 K .

Table -4.

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$$[\text{BPBC}] = 10^{-3} \text{ mol dm}^{-3} \quad [\text{HClO}_4] = 0.1 \text{ mol dm}^{-3} \quad [\text{Micelles}] = 1.00 \times 10^{-2} \text{ mol dm}^{-3}$$

$$[(\text{NH}_3)_5\text{Co(III)-L}] = 1.00 \times 10^{-2} \text{ mol dm}^{-3} \quad \text{Temperature} = 55 \pm 0.2^\circ\text{C}$$

10 ² [(NH ₃) ₅ Co(III)-L] mol dm ⁻³	10 ⁴ k ₁ (s ⁻¹)								
	NaLS	TRITON	CTAB	NaLS	TRITON	CTAB	NaLS	TRITON	CTAB
	Co ^{III} Thio Glycolato			Co ^{III} Thio Malato			Co ^{III} Thio Lactato		
0.5	1.617	2.268	2.562	1.904	2.374	2.771	2.157	2.502	2.967
1.0	3.237	4.538	5.125	3.804	4.690	5.437	4.312	5.006	5.937
1.5	4.857	6.806	7.829	5.758	7.112	8.371	6.471	7.514	8.904
2.0	6.472	9.064	10.434	7.575	9.536	11.061	8.632	10.004	11.876
2.5	8.098	11.348	13.040	9.508	11.852	14.099	10.795	12.520	14.835

Figure 3 shows the plot of log of Co (III) complexes α -Thio acids versus log k₁ for the oxidation of Co (III) of α -Thio Lactato, α -Thio Malato and Co (III) of α -Thio Glycolato by BPBC in the presence of CTAB at 328K. The rate of oxidation increased progressively with increasing the concentration of Co (III) Complexes of α -Thio acids. The slope is 0.999 measured.

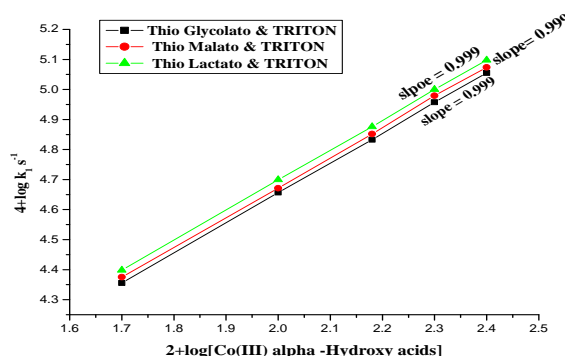


Fig 3

6.5. Mechanism

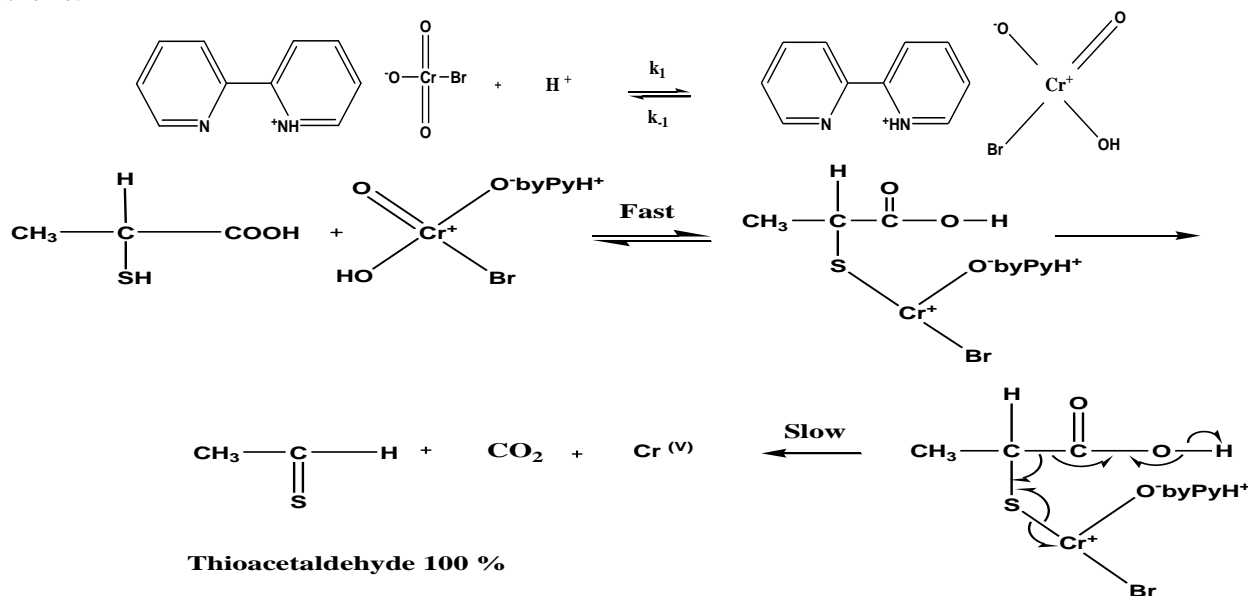
The oxidation of alpha-Thio acids and their complexes by BPBC in an atmosphere of nitrogen failed to induce the polymerization of acrylonitrile which discounts the possibility of any radical formation. Oxidation of alpha – Thio acids by 2,2'-bipyridinium bromo chromate under followed pseudo first order reaction. The oxidation of alpha-Thio acids and their complexes was catalysed by perchloric acid, which may well be attributed to the protonation of BPBC to give a stronger oxidant and electrophile. The kinetic results and absence of keto acids as a product leads to a mechanism involving one electron transfer to Cr (VI) with C-C cleavage (scheme 1). In the oxidation of the complexes, the available evidence point out the absence of two electron transfer to Cr (VI) and C-H cleavage. Hence, a mechanism involving one electron transfer to Cr (VI) and an induced electron transfer to Co (III) by the decarboxylation and synchronous cleavage of C-C was proposed. The complexes cobalt (III) reduction in to cobalt (II).The product was formed Thio acetaldehyde (100 %) Scheme 2.

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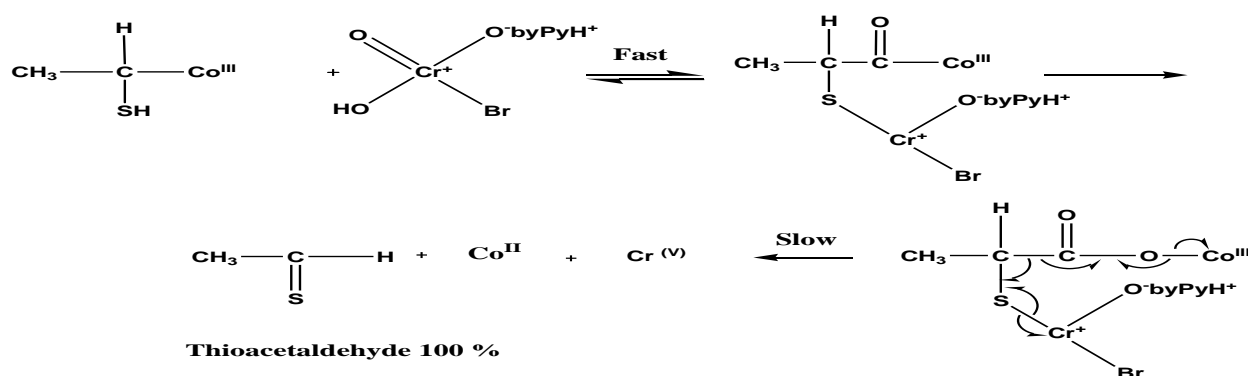
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Scheme: 1



Scheme: 2



VII. CONCLUSION

The kinetics and oxidation cobalt (III) of alpha Thio acids viz, Thio glycolic Thio Malic acid Thio Lactic acid acid, by 2,2'-bipyridinium bromo chromate in surfactant medium have been using a novel chromium (VI) oxidant. The rate of the reaction increase the observed presence surfactant NaLS, CTAB and TRITON-X 100. The comparison of the rate of cobalt (III) complexes bound Thio acids much more than the cobalt (III) complexes unbound Thio acid with micelles. Among the surfactant which CTAB enhances the rate much more than the TRITON-X 100, NaLS. The temperature increases the rate of the reaction increases with micelles CTAB higher than the TRITON, NaLS. Mechanism explains the synchronous C-C bond fission and electron transfer to cobalt (III) centre.

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