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ISSN 2349 - 5138

E - ISSN 2348 - 1269

An open Access, peer reviewed, refereed, online and print research journal

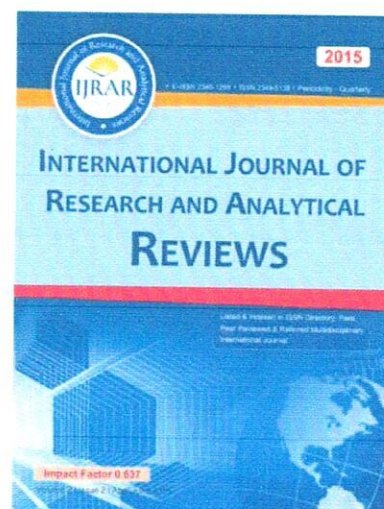
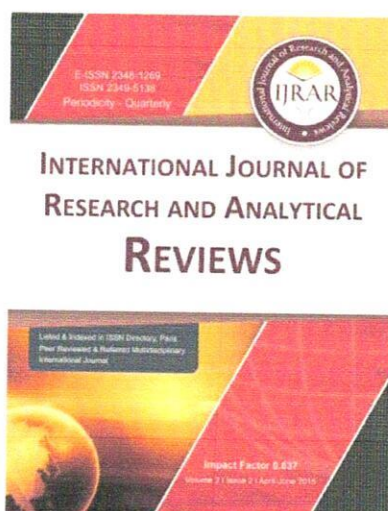
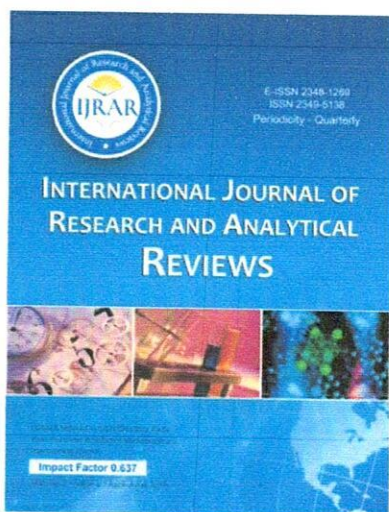
International Journal of Research and Analytical Reviews

UGC Approved Research Journal

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2061-C/2/B, Nr. Adhyatma Vidya Mandir, Sanskar Mandal, Bhavnagar-364002.

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KINETIC STUDY OF OXIDATIVE DEGRADATION OF P-CHLORO-SUBSTITUTED AROMATIC CARBOXYLIC ACID HYDRAZIDE BY VANADIUM (V)

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ABSTRACT: Chemical kinetic study of oxidative degradation of p-chloro- substituted aromatic carboxylic acid hydrazide by Vanadium (v) was studied in aqueous ethanol medium under pseudo first order condition. The formation of complex between the reactants, which decomposes in the subsequent step to give corresponding aromatic carboxylic acid. Transfer of single electron with intervention of free radical was observed during the course of the reaction. The effect of different initial $[V(v)]$ on reaction rate shows constancy in k values which indicates the, pseudo-first order kinetic behavior of the reaction. Increase in hydrazide concentration decreases the specific rate. The rate of reaction is directly proportional to the Concentration of the acid as well as dielectric constant of the medium of the reaction. There is no effect of change in Concentration of salt as well as various salts of the same concentration under the experimental conditions on the reaction rate.. The variation of ionic strength during the course of the reaction has negligible effect on rates of reactions. The study of effect of temperature on the rate of the reaction was done in between 30 to 55 °C and the specific reaction rates are found to be directly proportional to the increase in temperature.. The activation parameters were determined and the values support the proposed mechanism as evidenced by considerable decrease in entropy of activation. ($-\Delta S^\ddagger = 144.98, J K^{-1} mol^{-1}$). The progressive study of the reaction was followed by measuring the absorbance (A) of the $V(v)$ at 410nm.

Keywords: p-chloro- substituted aromatic carboxylic acid hydrazide, Ammonium metavanadate, Oxidative degradation, pseudo-first order, Thermodynamic parameters.

I. Introduction

Hydrazides are obtained by preparing derivatives of Carboxylic acids and hydrazines¹. It's multiple use in various branches of chemistry^{3,4,5,6,7,8} especially pharmaceutical Chemistry², needs to study the mechanism of their oxidation in detail. The process of oxidation reaction is an important transformation in an organic chemistry. The formation of corresponding acids^{9,10,11,12,13,14,15,16} is observed during the most of the oxidation reactions of hydrazine's. Literature survey shows that, the chemical kinetic study of oxidative degradation of substituted aromatic carboxylic acid hydrazides by vanadium (v) is not extensively studied. Therefore kinetic study of p-chloro- substituted aromatic carboxylic acid hydrazide by vanadium (v) was undertaken. The study of Chemical kinetics deals with the rate at which the chemical reactions occur and the influence of various factors such as concentration, temperature, pressure catalysts etc. on the reaction rates. Different chemical reactions occur at different rates.

II. MATERIAL AND METHOD

The anhydrous $NaHCO_3$, H_2SO_4 , salt, ammonium metavanadate (AR grade), hydrazine hydrate (BDH 99%) and fresh distilled ethanol was used. Ethyl ester of p-Chloro substituted aromatic carboxylic acid was prepared by esterification¹⁷ and converted to corresponding hydrazide by using the prescribed procedure¹⁸. An equimolar mixture of ethyl ester of p-Chloro substituted aromatic carboxylic acid and hydrazine hydrate (B.D.H. 99%) was refluxed for 15 minutes. Then enough absolute ethanol was added through the condenser to get clear solution and further refluxed for more than six hours. The excess of hydrazine hydrate, solvent ethanol and other unreacted material was removed by distilling the solution under reduced pressure. The resulting hydrazide recrystallised from ethanol and stored in amber colored bottles and kept in dark place. The distilled water obtained by redistillation of distilled water in the presence of a few crystals of $KMnO_4$ and a few pellets of KOH using Borosil glass distillation assembly was used throughout the experiment. The 0.01 M solution of oxidant ammonium metavanadate was prepared by dissolving accurately calculated and weighed quantity of ammonium metavanadate in hot double distilled water using Pyrex glass measuring flask. The standardization of $V(v)$ solution was done by titrating it against standard $Fe(NH_4)_2SO_4$ solution by using diphenylamine as an indicator. Similarly the stock solution

of NaClO₄ was prepared by dissolving equivalent quantities of Na₂CO₃ and HClO₄(70% E.Merck) in H₂O to maintain ionic strength. Standard p-chloro-benzoic acid hydrazide solution was prepared by dissolving it in aqueous alcoholic medium.

2.1 Determination of λ max for the reaction:

The absorption maxima ie λ max of the oxidation reaction under study was determined by varying wavelength, it was observed that maximum absorbance for both ammonium metavanadate and a mixture of hydrazide and ammonium metavanadate was obtained at 410 nm λ max.

2.2 Method of following the kinetic reaction:

The oxidant and substrate are taken in separate conical flasks along with required quantities of sulphuric acid and sodium perchlorate and are kept in a thermostat at 35 \pm 0.1 $^{\circ}$ C for more than half an hour. The kinetic study was followed by mixing thermally equilibrated solution of reactants and transferring the reaction mixture to 1cm³ cuvette. The progress of reaction was followed by measuring absorbance of the reaction mixture at 410 nm spectrophotometrically using UV-Vis. Spectrophotometer in sulphuric acid medium using water as a reference solvent. The reaction was studied under pseudo-first order condition in which, concentration of hydrazide was in excess as compared to that of ammonium metavanadate. The reaction is found to proceed through formation of complex between vanadium (v) and hydrazide. The pseudo-first order rate constant K was obtained by plotting the log of absorbance at 410 nm against time for hydrazide and was found to be fairly constant at different concentrations of vanadium (v).

2.3 Reaction intermediates:intervention of free radical:

The induced polymerization of acrylonitrile¹⁹ and spontaneous reduction of mercuric chloride²⁰ in reaction mixture itself indicates that, the present reaction involves the formation of free radical.

2.4 Effect of sulphuric acid on hydrazides:

A standard kinetic experiment was repeated by only changing the concentration of H₂SO₄ from 0.0025 to 0.1 M containing equivalent quantities of hydrazides, oxidant, ionic strength, salt and temperature. The mixtures in each flasks were made alkaline and tested for detection of hydrazine after three hours. It was seen that, the resultant solution in various flasks didn't give any response to ammonical AgNO₃ and Fehling's solution, indicating absence of hydrazine. Hence it can be concluded that, there is no significant hydrolysis of hydrazide in the presence of H₂SO₄ up to 0.1 M.

2.5 Alternative method for confirmation of status of acid content and to check hydrolysis of hydrazide under experimental conditions:

The confirmation of hydrolysis of hydrazide under experimental conditions was done by conducting similar set of experiments. It was subjected to alkalimetric estimation of acid content in the beginning and at the end; when it was found that there was no perceptible change in acid content of any mixture after a period of minimum three hours. This invariance of acid concentration can be attributed to the fact that; there was no hydrolysis of hydrazide under experimental conditions was observed. Similar observations are already reported.²¹

2.6 Determination of stoichiometry of the reaction:

The number of moles of V(v) consumed by one mole of hydrazide in oxidation reaction under investigation means the stoichiometry of the reaction was determined by spectrophotometric method. A set of five reaction mixtures containing a known excess of V(v) over hydrazide in the presence of 1.0 x 10⁻² M H₂SO₄ and 5.0 x 10⁻² M NaClO₄ was kept in a thermostat at 35 $^{\circ}$ C for more than 48 hours. A blank experiment without hydrazide was carried out concurrently using identical quantities of V(v), H₂SO₄ and NaClO₄ after volume correction with water for hydrazide solution.

After completion of the reaction, concentration of V(IV) was determined at 765nm (ϵ =17.77).²² from its absorbance and molar extinction coefficient (ϵ) by using the expression $C = \text{Abs.} / \epsilon$

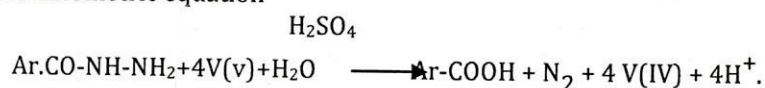
Finally, the moles of V(IV) formed in oxidation of one mole of hydrazide were calculated in each case by using the relation. Mole ratio = - [V(IV)] / [Hydrazide]₀

The value of [V(IV)] / [Hydrazide]₀ for different experiments was found to be nearly equal to 4. Therefore it is concluded that 4 moles of ammonium metavanadate were required for oxidation of one mole of hydrazide.

2.7 Identification of oxidation products:

For the identification of oxidation products; the reaction was studied by using hydrazide and ammonium metavanadate in stoichiometric proportions in presence of 1.0 x 10⁻² M sulphuric acid and 1.0 x 10⁻¹ M sodium perchlorate. The flask containing reaction mixture was kept in a thermostated water bath maintained at 35 $^{\circ}$ C for 24 hours to complete the reaction. After the completion of the reaction, the reaction

mixture was subjected to ether extraction and acid was separated. The presence of carboxylic acid group was detected by testing with 5 percent bicarbonate solution. The amide derivative of the corresponding aromatic carboxylic acid was prepared²³. The observed physical constant of amide derivatives was found to be in good agreement with those of benzamide. The formation of corresponding carboxylic acids in oxidation of hydrazides was accompanied by evolution of nitrogen gas. Nitrogen was detected by lime test.²⁴ A mixture of lime and manganese dioxide in 10:1 proportion was ignited in a small hard glass tube. A test portion of concentrated reaction mixture was rendered neutral with sodium hydroxide solution and it was added to the ignited mixture. The tube was heated slowly and the liberated gas was tested with filter paper moistened with manganese nitrate and silver nitrate solutions. This indicator paper held at the mouth of the tube shows grey fleck, which turns blue immediately on treatment with a drop of benzidine solution which, indicates the formation of nitrogen gas during the oxidation. Finally considering the products of oxidation, observed mole ratio and material balance, the oxidation of hydrazide can be represented by the stoichiometric equation



The above equation not only consistent with stoichiometry and products of reaction, but also it explains the observed gradual decrease in k values with increase in time, which can be attributed to increasing proportions of H⁺ ions with the progress of the reaction.

III. RESULTS AND DISCUSSION:

It was found that, the reaction proceeds with a measurable velocity using 5.0×10^{-4} M ammonium metavanadate, 5.0×10^{-3} M Substrate hydrazide, 1.0×10^{-1} M salt sodium perchlorate, 1.0×10^{-2} M sulphuric acid and 410nm λ_{max} at 35°C. The observed rate constant (k) of the reaction goes on slightly decreasing with time, the order of reaction with respect to V(v) is one.

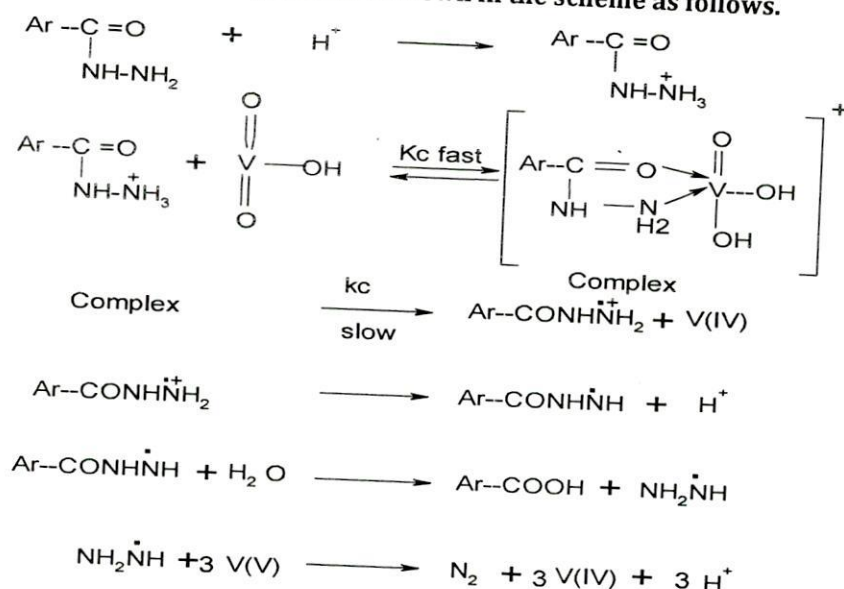
Table 1: The specific reaction rate constants determined by graphical method are:

1	Effect of	Unit Concentration	Multiples of Unit Concentration and respective rate Constants						
			1.0	2.0	4.0	5.0	6.0	8.0	10
1	[hydrazide]	[p-Cl-BAH] x 10 ³ M	1.0	2.0	4.0	5.0	6.0	8.0	10
		k x 10 ⁴ sec ⁻¹	3.59	2.69	1.59	0.989	0.645	0.346	0.189
2	[AMV]	[AMV] x 10 ⁴ M.	1.0	2.0	4.0	5.0	6.0	8.0	10.0
		k x 10 ⁴ sec ⁻¹	1.01	1.03	0.996	0.989	1.02	1.00	0.990
3	[H ₂ SO ₄]	[H ₂ SO ₄] x 10 ² M.	0.5	1.0	2.0	3.0	4.0	5.0	--
		k x 10 ⁴ sec ⁻¹	0.759	0.989	2.01	2.21	2.40	2.69	--
4	[NaClO ₄]	[NaClO ₄] x 10 ¹ M.	0.5	1.0	2.0	3.0	4.0	5.0	--
		k x 10 ⁴ sec ⁻¹	0.974	0.989	0.984	1.01	0.992	0.996	--
5	various Salts	[SALT] ₀ = 1.0 x 10 ⁻¹ M	LiCl	NaCl	KCl	LiClO ₄	NaClO ₄	KClO ₄	MnCl ₂
		k x 10 ⁴ sec ⁻¹	1.01	0.997	1.03	0.982	0.989	1.02	1.01
6	Dielectric Constant	Ethanol %	50	60	70	80	--	--	--
		Dielectric constant	64.92	59.98	54.65	47.78	--	--	--
		k x 10 ⁴ sec ⁻¹	1.29	0.989	0.797	0.629	--	--	--
7	Temperature	Temperature in °C	30	35	40	45	50	55	--
		k x 10 ⁴ sec ⁻¹	0.599	0.989	1.31	2.01	2.46	3.44	--

Table 2: The various Energy parameters calculated are:

Sr	Energy parameters	Value	Sr	Energy parameters	Value
1	Temperature Coefficient	1.89	4	Entropy of activation ΔS^\ddagger (J K ⁻¹ mol ⁻¹)	-144.98
2	Energy of activation E _a (KJ mol ⁻¹)	55.97	5	Free energy of activation ΔG^\ddagger (KJ mol ⁻¹)	98.89
3	Enthalpy of activation ΔH^\ddagger (KJ mol ⁻¹)	45.92			

Mechanism of the reaction: The detailed mechanism of the reaction in terms of the active species of the HVO_3 and substrate protonated hydrazide is shown in the scheme as follows.



where, $\text{Ar} = \text{C}_6\text{H}_4\text{Cl}$

According to the above scheme the rate of the reaction is given by

$$\text{Rate} = k_c [\text{Complex}]$$

Substituting the value of [Complex] from the equilibrium,

$$\text{Rate} = k_c K_c [\text{HVO}_3] [\text{Ar}-\text{CONH}\text{NH}_3^+].$$

SCHEME

IV. CONCLUSION:

The investigation of the effect of hydrazide concentration on the reaction rate shows that, The pseudo-first order rate constant decreases with increase in hydrazide conc. The decrease in rate constant as the concentration of hydrazide increases can be attributed to greater stability of the complex in alcoholic medium probably due to solvation²⁵.

The effect of oxidant concentration on the reaction shows that, the rates of oxidation of hydrazide under examination are almost constant with increase in concentration of $\text{V}(\text{v})$ and the order of reaction with respect to vanadium (V) remains one throughout the used concentrations of vanadium (v). In other words, Oxidation follows the first order kinetics because the $\log(\text{Abs})$ versus time plot in each case is linear with positive slope and intercept on $\log(\text{Abs})$ axis. The constancy of k values at different initial $[\text{V}(\text{v})]$ indicate the pseudo-first order kinetic behavior of reaction.

The effect of concentration of sulphuric acid on the rates of oxidation of hydrazide under study shows that, the specific reaction rate increases as the concentration of acid increases.

The reaction rates are not influenced by increase in ionic strength similarly the reactions rates are not influenced by using various salts, under the experimental conditions.

The specific rate of reactions is decreasing with decrease in dielectric constant²⁶.

The study of effect of temperature on oxidation of hydrazide clearly shows that, rate of reaction depends on temperature and it increases with increase in temperature. The values of observed rate constants were used to determine various thermodynamic parameters like temperature coefficient(189), energy of activation (E_a) $55.97 \text{ KJ mol}^{-1}$, enthalpy of activation (ΔH^\ddagger) $45.92 \text{ KJ mol}^{-1}$, entropy of activation (ΔS^\ddagger) $-144.98 \text{ J K}^{-1} \text{ mol}^{-1}$ and free energy of activation (ΔG^\ddagger) $98.89 \text{ KJ mol}^{-1}$. The $\log(\text{Abs})$ against time plots at different temperatures are linear which reveals that, the pseudo-first order kinetic behavior of the reaction is not affected by change in temperature. The formation of free radicals or radical ions during the course of reaction was confirmed from induced polymerisation of acrylonitrile^{19,20}. The formation of carboxylic acids and N_2 in the oxidation of aliphatic as well as aromatic acid hydrazides²⁸ is well documented in chemical literature. The mole ratio of hydrazide:vanadium (v) is found to be 1:4 and it is independent of concentration of sulphuric acid that was used.

The integer value of observed mole ratio, its independence on sulphuric acid concentration and formation of only carboxylic acid along with nitrogen gas as oxidation products leads to deduce that, the two rate determining steps occurring simultaneously results in the formation of one and the same intermediate. Although the observed mole ratio (substrate : oxidant) of the reaction is 1:4, as is pointed out earlier, the order of reaction with respect to vanadium (v) is one.

V. Acknowledgement:

Author expresses his sincere thanks to the principal of Bharati Vidyapeeth's Matoshri Bayabai Shripatrao Kadam Kanya Mahavidyalaya Kadegaon for providing necessary facilities during the course of this research work.

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 Impact Factor Calculation By Google Scholar h-index

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 WE Count our Impact Factor based on Google Scholar h-index impact factor of 2018: 5.75 as per h-index calculation

JETIR 6 Year impact factor calculation based on Google Scholar h-index

A = Total number of Google scholar citation in YEAR 2014,2015,2016,2017,2018,2019

B= Total number of Article Published in YEAR 2014,2015,2016,2017,2018,2019

C= Subset of citation and h index is subset of A

D= B/C Total impact factor

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