

CHLORINATION OF N-BENZOYL VALINE BY SODIUM N-CHLORO-PARA-TOLUENE SULFONYL AMIDE (CAT) HYDROCHLORIC ACIDIC: A KINETIC AND MECHANISM STUDY

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Abstract. The mechanism of N-benzoyl valine oxidation by chloramine-T in hydrochloric acid medium at temperatures ranging from 308 to 328 K is the subject of this research. The findings of the experiment showed that the medium's ionic strength had an influence, as the affixing of P-toluene sulphone amide slightly retarded the reaction, and that the reaction was first order with regard to the substrate and chloramine-T. Based on kinetic investigations, the reaction ratio 1:1 is suggested.

Keywords: Chlorination, N-benzoyl Valine, (CAT), kinetic.

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1. Introduction

Kinetic studies have been conducted on the mechanism of many oxidation processes of sodium N-chloro-4-methyl benzene Sulfonamide $p\text{-CH}_3\text{-C}_6\text{H}_4\text{SO}_2\text{NCINa}\cdot 3\text{H}_2\text{O}$, also recognized as (CAT) chloramine-T; under both acidic and alkaline condition, it acts as an oxidizing agent, producing sodium chloride and p-toluene sulphone amide. Chloramine-T provides a number of active species, including RNHCl , HOCl , and RNCl , OCl^- ions in an alkaline medium and DCT (Dichloramine-T) in an acidic solution [1]. The type of oxidized material determines which of these components is more effective, and altering the experimental setup typically increases the effectiveness of one component over the other. Some research has studied the behavior of using chloramine-T as an oxidant [2-4]. The Ir(III) catalyst is used for oxidation of Leucine [5] L-Alanine [6], mannitol [7], and Fructose [8] by chloramine-T. Several kinetics investigations have been carried

out with this oxidizing agent such as oxidation of some aromatic attic primary diamines in the presence of HCl [9], thiourea and N-substituted thiourea [10], amide [11], carboxylic acid [12], alcohol [13], tetrahyrofuran [14], ether [15] and chlorination of N-acetyl glycine [16] and alanine [17]. Due the amino acids are essential for metabolism in addition to being building blocks for protein synthesis, amino acid oxidation is a biological process of great interest. The kinetics and mechanism of amino acid oxidation in various applications in biochemical research have been studied by many researchers [18]. Furthermore, amino acid oxidation is interesting because different oxidants lead to the formation of different products one of the biochemical progresses. Using various oxidants amino acid oxidation and kinetics have been extensively studied. On the other hand, the literature on N-benzoyl valine oxidation has relatively few reports.

2. Experimental Part

Materials. All chemicals that were used in the manuscript were produced by Alpha Acer BDH and Sigma Aldrich companies. A spectrophotometer Double beam -

Spectrophotometer Shimadzu UV-1800 PC, UV-Visible was used.

Methods.

Synthesis of Chloramine – T [19, 20].

The procedure was used to prepare and purify chloramine-T A chloramine-T aqueous solution. It is kept in stoppered amber bottles and is standardized using the iodometric method.

Synthesis of N- benzoyl valine [21, 22].

Dissolving 1 mole of the amino acid (valine) in the solution of 10% NaOH and cooling at -5 to -10 °C, added 1 mole of benzoyl chloride

gradually with continuous stirring and finally the medium was neutralized, the white precipitate was filtered and dried. Obtained a yellow powder with a yield of 78%, and m.p. (130-132) °C.

IR (cm⁻¹): 3323 (N-H), 3070 (C-H_{arm}), 2995, 2826 (C-H_{alph}), 1748 (C=O) acid, 1678 (C=O) amide, 1275 (C-O) acid (Fig.1).

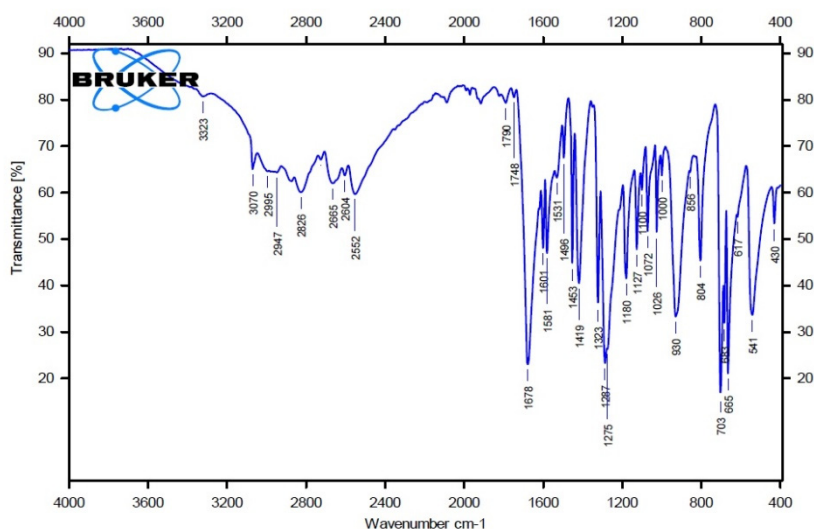


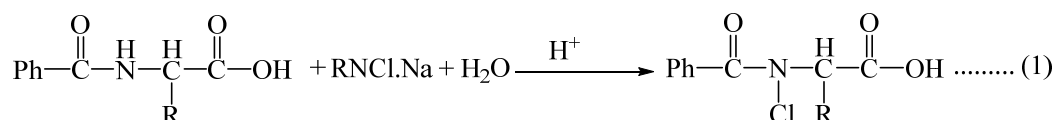
Fig.1. IR spectrum for prepared compound (N-B-V)

Kinetic measurements [23, 24]. Pseudo first-order conditions were used to conduct the reactions in Pyrex at 308 K using a known substrate excess over an oxidant. The necessary quantities of substrate solutions for each run. To maintain a constant total volume during all runs, HCl, NaClO₄, and water were placed within the tube and thermostat at 308 K. The measured amount of CAT solution was thermostat at the same temperature. Following the reaction, 2 ml of the mixture was taken out and added to a flask including potassium iodide and 2M of sulfuric acid to quench the reaction.

Iodine that was titrated against a

conventional sodium thiosulphate solution as titrant using starch as an indicator is liberated when the unreacted chloramine-T rapidly reacts with the KI solution. after this reaction, the free iodine was measured spectrophotometrically at a wavelength of 353 nm.

Stoichiometry. Different ratios of CAT to N-benzoyl Valine were equilibrated in the Company of 0.2 (M) of HCl, water, and ethanol, at 308 K for 24 hours. Determination of the remaining Oxidant demonstrated that 1moles of chloramine-T was consumed Per mole of N-benzoyl Valine the chemical measurements obtained can be represented as follows:



p-toluene sulfonamide was detected Among the products of the reaction using paper solvent by chromatography [25], The resulting N-chlorobenzoyl valine was identified as an alight crystal by infrared spectroscopy (cm⁻¹): 3063

(C-H_{arm}), 2922 (C-H_{alph}), 1741 (C=O) acid , 1655 (C=O) amide , 1300 (C-O) acid In addition to (812,659) cm⁻¹ of the (N-Cl)bond [26], Note that the stretch band (NH) has disappeared (Fig.

2) at m.p. (106-108) °C and from chlorine elemental analysis testing.

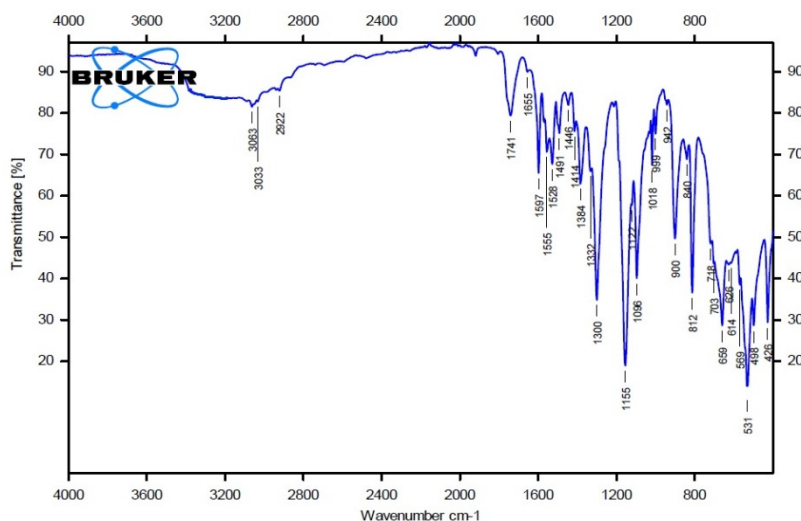


Fig. 2. IR spectrum for product chlorination compound (N-B-V)

3. Results and Discussion

At various initial reactant concentrations kinetics of the chlorination of N-benzoyl valine by CAT has been investigated in the hydrochloric acid media at 308 K.

The effect of different concentrations of reactants on the reaction rate. Plotting the log CAT versus time at constant acid concentration

and excess substrate concentration was linear ($r > 0.9954$) (Fig.3), It suggests that [CAT] has a first-order rate dependence. The pseudo-order of rate constant is included in Table 1 the values of which are unaffected by increasing substrate concentration, showing that the [N-B.V.] concentration had no effect on the reaction rate.

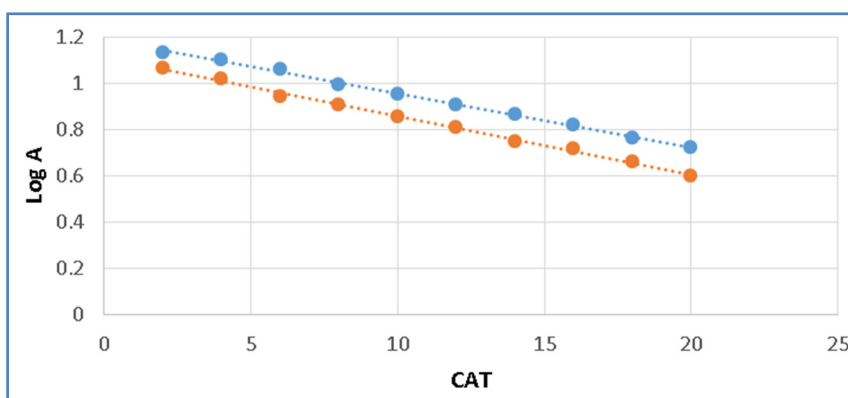


Fig. 3. Graph log [CAT] versus Time for oxidation of N-benzoyl Valine at 308 K.

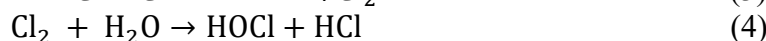
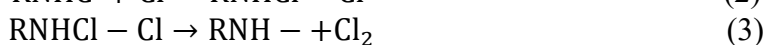
Table 1. The effect of variation [N-B.V.] and [CAT] concentration at 308 K, [HCl]=0.05M, M=0.025 M, 25% ethanol.

10^3[CAT] M	10^3[S] M	$10^4 k \text{ Sec}^{-1}$
1.50	0.75	9.7871
1.50	1.00	9.8640
1.50	1.25	9.8265
1.50	1.50	9.7871
1.50	1.75	9.8265
1.25	1.00	11.9754
1.50	1.00	9.8640

1.75	1.00	8.6743
2.00	1.00	7.6765
2.25	1.00	6.4480

The effect of different concentrations of hydrogen and chloride ions. On varying the concentration of hydrochloric acid from (0.03-0.11) M Table 2, it was observed that an increase in reaction rate when increasing the concentration of hydrogen ion. Graphing log k against $[H^+]$ got a straight line (Fig. 4). Consequently, the first-order reliance on hydrogen concentration is shown, while the rate constant of reaction will intensify slightly with a

rise in $[Cl^-]$ concentration (Table 2). Plotting the log k versus $[Cl^-]$ gives straight line with an 0.2 slope. The increase the rate constant is the creation of HOCl as a result of the way in which the chloronium and chloride ions interact generated by the N-Cl bond's hydrolysis accelerates the rate of reaction. The reason for the increase in the rate of reaction is causes the formation of HOCl based on the reaction that follows [27].



Therefore increasing the concentration of chloride ions produces more the reaction's

active oxidizing species (i.e. HOCl), and as a result, the reaction rate is seen to increase [28].

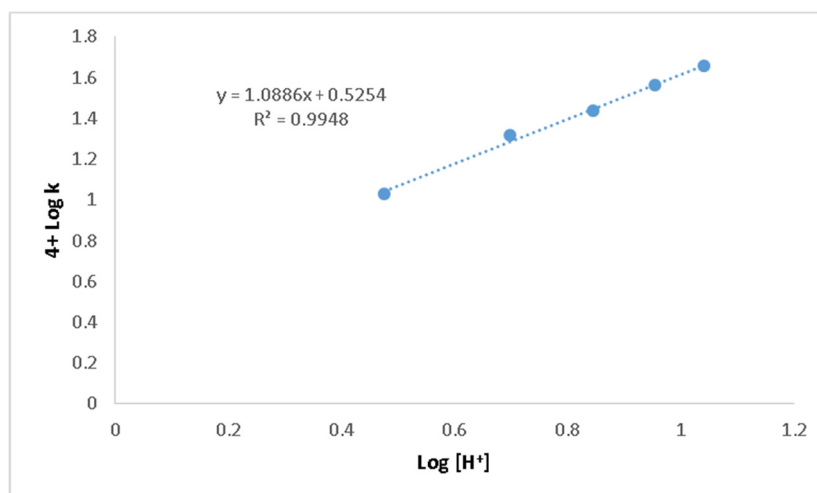


Fig. 4. Graph Log K Versus Log $[H^+]$ for oxidation of N-benzoyl Valine at 308 K

Table 2. Effect of variation of hydrochloric acid $[H^+]$ and Sodium chloride $[Cl^-]$ concentration at 308 K $[CAT]=0.0015$ M; $[N-B.V.] = 0.0$, $M=0.025$ M; 25% Ethanol

$10^2[H^+]$ M	$10^2[Cl^-]$ M	$10^4 k$
3.00	5.00	10.708
5.00	5.00	20.72
7.00	5.00	27.36
9.00	5.00	36.31
11.00	5.00	45.25
5.00	2.50	18.38
5.00	5.00	20.72
5.00	7.50	22.99
5.00	10.00	24.56
5.00	12.50	27.48

Effect of changing of the Ionic strength additional p-toluene sulphone amide. The rate of reaction was unaffected by incorporating concentrated sodium perchlorate solution to

change the medium of ionic strength (0.025-0.125M). Additionally, Table 3 shows that the addition of P-toluene sulphone amide (P-TSA) somewhat changed the rate of reaction [29].

Table3. Effect of variation of Product [P-TSA] and Sodium perchlorates NaClO_4 of [N-B.V.] at 308 K°; [N-B.V.] = 0.01 M, [CAT]=0.0015, [H]=0.05 M, ethanol 25%

$10^2[\text{NaClO}_4]$ M	$10^4 k_1$ Sec ⁻¹	$10^3[\text{p-TSA}]$ M	$10^4 k_1$ Sec ⁻¹
2.50	9.8640	1.00	9.5239
5.00	9.6431	2.00	9.2999
7.50	9.7851	3.00	8.9898
10.00	9.6134	4.00	8.7111
12.50	9.8123	5.00	8.3267

The effect of changing the medium's dielectric constant on the rate. Using ethanol (25%–55%) in the reaction solution changed the medium's dielectric constant (D), and the rate

constant's value increased as the ethanol amount increased (Table 4). Plots showing Log k versus I/D have a positive slope [30].

Table 4. Effect of varying the medium's dielectric constant on the rate reaction at 308 K, [N-B.V.] =0.01M, [CAT]=0.0015, [H]=0.05M

Ethanol V%	D	$10^4 k_1$ Sec ⁻¹
25.00	58.53	9.8640
35.00	53.22	11.2164
45.00	49.49	12.4365
55.00	43.97	13.6523
65.00	39.04	16.5412

The effect of temperature. The Arrhenius equation has been used to study the rate of reaction at various temperatures (308-323 K), and (Fig. 5) shows plotting of log k_1 vs $1/T$, it was observed that increasing the temperature increases the rate of reaction. activation energy

and its parameters of the compound were evaluated. These values are given in the Table 5. The values of activation enthalpy, entropy, and free energy suggest that the reaction's progress is not spontaneous [31].

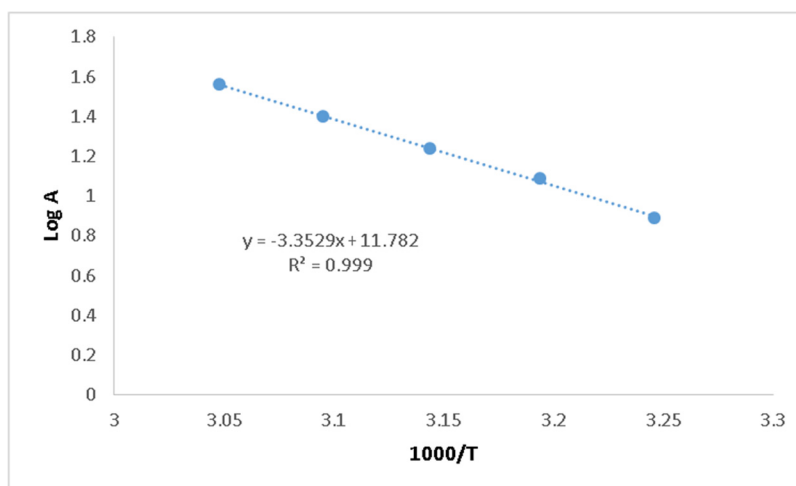


Fig. 5. Plotting Log k vs. $1/T$ of N-benzog Valine at Valine at 308 K

Table 5. Temperature's effect on reaction rate, [N-B.V.]=0.0M, [CAT]=0.0015M, [H⁺] = 0.05 M =0.025

T; K	10 ⁴ k ₁ ; Sec ⁻¹	ΔH [‡] ; kJ/mole	ΔS [‡] ; J/(mole·K)	ΔG [‡] ; kJ/mole	Log A; Sec ⁻¹
308	9.8640	53.96739	-288.333	88.86068	7.581169
313	14.0860	53.92582	-307.316	96.24398	6.582569
318	18.5001	53.88425	-307.754	97.91981	6.552806
323	28.1101	53.84268	-306.898	99.182	6.590754
328	38.3440	53.80111	-306.858	100.7032	6.586187

$$E_a = 2.9871 \times 10^3 \text{ kJ/mole}$$

$$\Delta H^\ddagger (\text{average}) = 53.8842 \text{ kJ/mole}$$

$$\Delta S^\ddagger (\text{average}) = -303.432 \text{ J/(mole·K)}$$

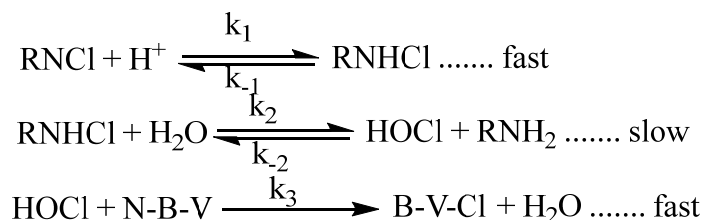
$$\Delta G^\ddagger (\text{average}) = 96.5819 \text{ kJ/mole}$$

$$\text{Log A (average)} = 6.7786 \text{ Sec}^{-1}$$

Discussion. Chloramine-T was used as a moderate oxidizing agent in both alkaline and acidic conditions. In general, two electrons are exchanged in chloramine-T reactions to produce p-TSA (p-CH₃-C₆H₄SO₂NH₂) and NaCl, which are reduction products. The chloramine-T redox potential is According to the medium's PH, the p-TSA redox couple [32, 33] changes 0.778 V at pH 7.0, 0.614 V at pH 9.7, and 1.139 V at pH 0.65. The aqueous solution of CAT exhibits significant electrolyte properties. Depending on the pH value, chloramine-T generates various reactive species [34]. RNCl₂ (dichloramine-T), HOCl, and RNHCl are examples of potential oxidizing species of chloramine-T in acidic solution. Based on CAT, the rate law predicts a second-order dependent if RNCl₂ a reactive species, which is at contrasts with the actual results. Chloramine-T reactions in solutions include the presence of polar molecules and ions. It has been found that when the proportions of the solvent components change,

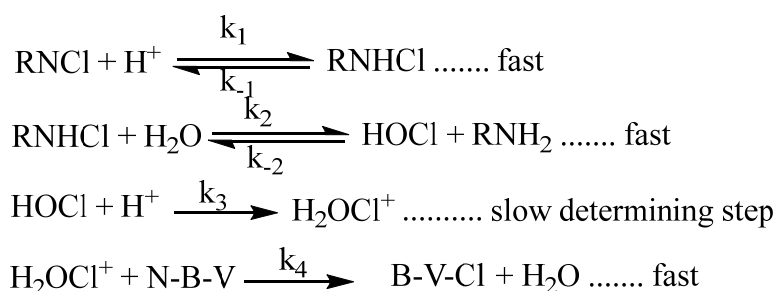
the reaction rate will change and that the relationship between the log k versus 1/D will give a straight line with a positive slope, meaning the participation of a positive ion with a dipole molecule in the rate determining step of the reaction. It has been observed that the reaction rate does not change when the ionic strength of the solution increases when different concentrations of sodium perchlorate are added. This indicator confirms the participation of a positive ion and a dipole molecule. It has been shown from the values of the reaction rate constants in Table 5 when the chloramine-T oxidizing agent reacts with N- benzoyl valine, the existence of two possibilities for the reaction will be expected [35].

The first possibility is that the active part of the chloramine-T is the positive ion, which is represented by (Cl⁺, H₂OCl⁺), while n-benzoyl amino acids will be a dipole molecule (Scheme I):

**Scheme I.**

As for the second possibility, the active part of Chloramine-T is the positive ion which is represented by H₂OCl⁺ as a dipolar molecule

with positive ion of n-benzoyl valine (Scheme II):

**Scheme II.**

Causes the gradual formation of HOCl through the hydrolysis of RNHCl. Rapid reaction with N-benzoyl valine comes next (Scheme I, step 3). Alternatively, it can undergo protonation to produce H_2OCl^+ (Scheme II, step

3), which interacts quickly with a substrate.

Using the steady state estimate in relation to HOCl and CAT, the following equation is obtained:

$$\frac{-d}{dt}[\text{CAT}] = \frac{K_1 K_2 [\text{H}_2\text{O}]}{K_{-1} + K_2 [\text{H}_2\text{O}]} [\text{CAT}][\text{H}^+] \quad (5)$$

this equation becomes

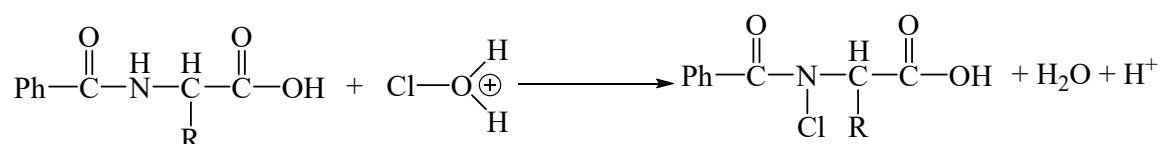
$$\frac{-d}{dt}[\text{CAT}] = [\text{CAT}] = K = [\text{CAT}][\text{H}^+] \quad (6)$$

Where:

$$K = \frac{K_1 K_2 [\text{H}_2\text{O}]}{K_{-1} + K_2 [\text{H}_2\text{O}]} = \text{constant}$$

Derivatization rates Law - equation (6) is complete based on experimental Kinetic study.

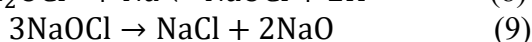
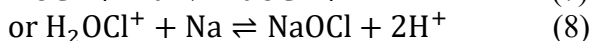
Scheme III provides the CAT's mechanism for oxidizing n-benzoyl valine [36]:

**Scheme III.**

This Scheme involves the removal of the provided n-chloro-acetyl Valine (the product) after an electrophilic attack by the partly cationic chlorine of H_2OCl^+ on the nitrogen atom of N-benzoyl Valine.

The first order constant somewhat

decreased when the initial chloramine-T concentration increased (Table 1), possibly as a result of deactivation carried on by the creation of small quantities [37] of NaClO_3 in side reaction giving further evidence that HOCl, H_2OCl^+ is of reactive type:



Lastly, the substantial negative value of the activation entropy, which denotes the production of a rigid transition state, is

supported by the proposed mechanism. Furthermore, the activator complex is more ordered than the reactant.

Conclusion

1. The rate of decrease in the concentration of chloramine_T per unit of time follows a first-order equation, as it was found that the rate of the oxidation of reaction depends on the concentration of chloramine_T and the hydrochloric acid.
2. When changing the ionic intensity by adding different concentrations of sodium perchlorate, it was found that the rate of the reaction rate is not affected by this change.
3. The rate constant of the oxidation reaction of N-benzoyl valine was slightly affected when different concentrations of the reaction product para toloune sulfonamate were added.
4. It was found that when the percentage of ethanol and water in the solvent increases, the rate of the reaction increases, i.e. in other words, the rate of the reaction increases with a decrease in the dielectric constant of the solvent.

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