

Mechanism of the Hydrolysis of α -(*p*-Nitrophenyl)cinnamonnitrile Derivatives¹

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Abstract—The rate constant hydrolysis of α -(*p*-nitrophenyl)cinnamonnitrile(NCPN) and its derivatives have been determined at various pH, and the rate equation which can be applied over a wide pH range is obtained. On the basis of the rate equation, hydrolysis product, general base, and substituent effects, a plausible mechanism of hydrolysis has been proposed: At pH < 4.0, the hydrolysis was initiated by the addition of water to β -carbon of the carbon–carbon double bond. At pH > 8.5, the addition of hydroxide ion to the double bond was rate controlling. In the range of pH 4.0–8.5, these two reactions occurred competitively.

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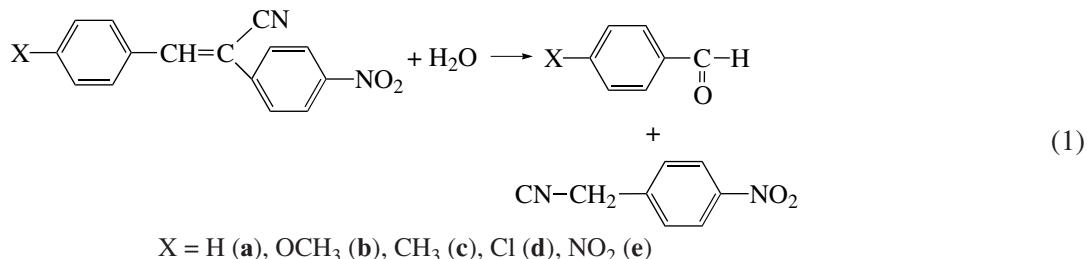
INTRODUCTION

A carbon–carbon double bond does not generally act as an acidic site. However, when the electron density of a carbon–carbon bond is reduced by strongly electron withdrawing substituents, nucleophilic attack at one of the vinylic carbons may occur. In addition to substituents and isomerizations, nucleophilic additions are one of the three major classes of reactions of carbon–carbon double bonds [1–7].

Extensive studies of nucleophilic addition to the double bond of activated olefins have led to a quantitative understanding of the relationship between the structure-reactivity and changes in the reactant structure or reaction conditions. Esterbauer et al. [9–10] prepared adducts from the reactions of many sulphydryl compounds with α,β -unsaturated carbonyl compounds. Kharash [11] and Hurd [12] et al. studied the mechanism for the addition of mercaptan to carbonyl compounds in basic media. Truce and coworkers [13] have studied the stereochemistry of the mercaptan addition reaction. The addition of thiols to α,β -unsaturated compounds is interesting because much infor-

mation has appeared in the literatures [14–17] concerning the antiviral and antitumor activities of their adducts. Walker and Young [18] determined the rate constants and activation energies for the hydrolytic decomposition of chalcone and six ring substituted mononitro derivatives. The hydrolysis of olefins which are activated by electron-withdrawing substituents has also been studied in 50% Me_2SO –50% water by Bernasconi et al. [19–23]. They have suggested a four-step mechanism involving a carbanionic intermediate or zwitterionic intermediate.

As shown in the above references, some kinetic studies for Michael addition and their related reactions in alkali solution have been presented. However, nucleophilic additions to alkenes in the neutral pH and acidic media have not been studied as much nor their mechanisms systematized as well as basic media. In the present study, it is attempted to determine each rate constant over a wide pH range and to reveal the reaction mechanism of the hydrolysis of α -(*p*-nitrophenyl)cinnamonnitrile, especially in the acidic media which has not been studied carefully before (Eq. (1)).



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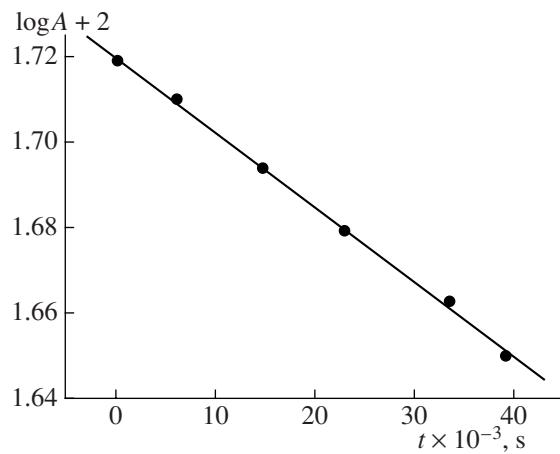


Fig. 1. Plot of log absorbance vs. time for the hydrolysis of NCPN at pH 3.0 and 40°C.

EXPERIMENTAL

General procedure. All of the reagents were commercially available and used without further purification. The ionic strength was kept constant to 0.1 M by adding sodium chloride except below pH < 1.0. Kinetic runs were obtained with a Shimadzu Spectrophotometer 210 A. IR spectra were taken with a JASCO A-210 infrared spectrophotometer. NMR spectra were obtained with a Bruker JYK 2C.100 NMR spectrometer (300 MHz). Elemental analysis was performed by an HP 185D CHN Analyzer, and melting points were measured with an Electrothermal IA 8100 digital MP apparatus. All of the buffer solutions were prepared from reagent grade chemicals. α -(*p*-nitrophenyl)cinnamonnitrile and its derivatives **1a–1c** were synthesized by reacting substituted benzaldehyde, *p*-nitrobenzyl cyanide, and sodium hydroxide in water as described previously [24, 25].

Product of hydrolysis. α -(*p*-Nitrophenyl)cinnamonnitrile (1.25 g, 5.0 mmol) was dissolved in 50 ml of a buffer solution (pH 9.0) of boric acid and sodium hydroxide containing 40% dioxane. The solution was stirred for 5 h at 50°C. The resulting solution was extracted twice with 50 ml of benzene. The solvent was evaporated, and the 2,4-dinitrophenylhydrazine was then added. After the remaining residue was acidified with 6 N HCl solution, the orange crystal was formed. The precipitate was filtered, washed twice with water, and dried in vacuo. The structural identity of the precipitate was confirmed by comparison of this data with reported data [26]. The melting point and IR spectral data of the crystal were consistent with the 2,4-dinitrophenylhydrazone ($C_6H_5CH=N-NHC_6H_3-2,4-(NO_2)_2$). Therefore, the reaction for the hydrolysis of NCPN produced benzaldehyde. mp 238°C (237°C [24]); IR(KBr) 1620, 1510 cm^{-1} .

Kinetics studies. Kinetics runs were made in water at 40°C. The α -(*p*-nitrophenyl)cinnamonnitrile was introduced as 1 ml of 2.0×10^{-3} M dioxane solution in

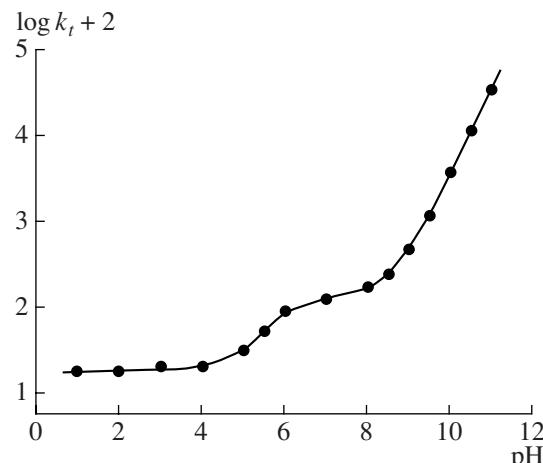


Fig. 2. pH-log(rate) profile for the hydrolysis of NCPN at 25°C. Circles are experimental points, and the solid line is drawn according to Eq. (10).

100 ml of final aqueous solution, and the flask was quickly shaken. Each aliquot (3 ml) of the solution was removed from the volumetric flask at time intervals and was placed in a quartz cuvette. The decrease of absorption at the wavelength of the maximum ($\lambda_{\text{max}} = 328-365$ nm) for the **1a–1e** with time was monitored.

Control experiments. The stability of **1a–1e** in dioxane solutions was determined by periodical scanning of the solution with the UV spectrophotometer. The solutions in dioxane were stable for at least one month when stored in the refrigerator.

RESULTS AND DISCUSSION

Determination of rate constants. Figure 1 shows the typical plot of logarithmic absorbance vs. time for the hydrolysis reaction of α -(*p*-nitrophenyl)cinnamonnitrile. The observed rate constant (k_{obs}) was estimated from the slope of plot log absorbance vs. time in Fig. 1.

Similar plots were obtained for the hydrolysis reactions of all compounds at all pH ranges (plots not shown). As shown in Fig. 1, the observed rate of **1a** was always of the first-order. The observed rate constant (k_{obs}) obtained from the slope at various pH are given in Table 1, and Fig. 2 shows the pH-log (rate) profile of this reaction.

The observed rate constants of the other derivatives were also determined by the same method (Table 2).

Substituent effect. The influence of the P-aryl substituent upon hydrolysis rates correlated satisfactorily with the Hammett equation using the values (Fig. 3). The Hammett ρ values were 0.8 and 0.47 for pH 3.0 and 9.0, respectively. This result indicates that the rate of hydrolysis is accelerated by electron withdrawing groups at all pH ranges. In addition, the observed ρ at pH 3.0 is larger than that calculated at pH 9.0. This result indicates less negative charge development in the reaction site of the transition state under basic conditions.

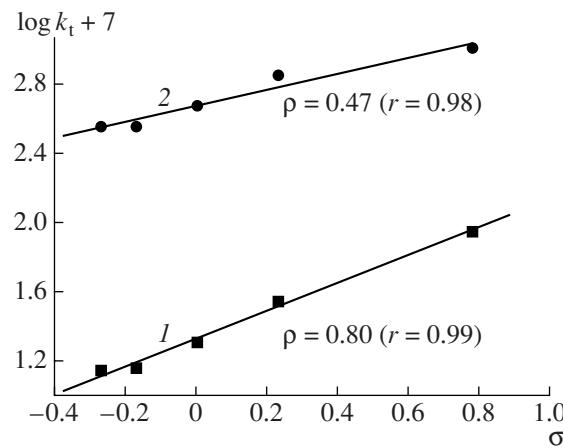


Fig. 3. Hammett plots for the hydrolysis of NCPN at (1) pH 3.0 and (2) pH 9.0 and 40°C.

General base catalysis. To make sure that this reaction is catalyzed by a general base, the rate constants were determined at various acetate ion concentrations at pH 4.78. As shown in Fig. 4, this reaction is catalyzed by a general base at $[\text{AcO}^-] < 0.1 \text{ M}$. However, as the amount of base becomes larger, the rate of hydrolysis approaches the limiting values.

Rate equation and mechanism. The hydrolysis reaction of **1a–1e** produced benzaldehyde derivatives

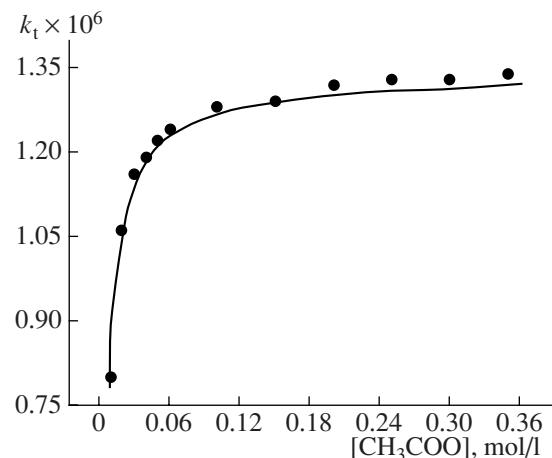


Fig. 4. General base catalyzed hydrolysis of NCPN at pH 4.78 and 40°C. Circles are experimental points, and the solid line is drawn according to Eq. (11-3).

and 4-nitrobenzyl cyanide as the products. The reaction is first-order to the substrate (Fig. 1), and the change of $\log k_t$ vs. pH is complicated (Fig. 2). From pH 1.0 to 4.0, the $\log k_t$ remains constant. However, from pH 4.0 to 8.5, the $\log k_t$ increases gradually, and above pH > 8.5, the slope is 1.0; i.e., k_t is directly proportional to the hydroxide ion concentration. A reasonable mechanism consistent with these results is shown in Scheme 1 and 2.

Table 1. Rate constants for the hydrolysis of NCPN at various pH and 40°C

pH	Buffer solution	$k_t \times 10^6, \text{s}^{-1}$	
		k_{obs}	k_{calc}
1.0	HCl	1.82	1.83
2.0	HCl	1.82	1.83
3.0	HCl	2.05	1.84
4.0	HOAc + NaOAc	2.07	1.98
5.0	HOAc + NaOAc	3.16	3.19
5.5	HOAc + NaOAc	5.25	5.26
6.0	HOAc + NaOAc	9.12	8.47
7.0	$\text{KH}_2\text{PO}_4 + \text{K}_2\text{HPO}_4$	12.6	12.9
8.0	$\text{H}_3\text{BO}_3 + \text{NaOH}$	17.3	16.9
8.5	$\text{H}_3\text{BO}_3 + \text{NaOH}$	24.3	25.0
9.0	$\text{H}_3\text{BO}_3 + \text{NaOH}$	48.4	50.1
9.5	$\text{H}_3\text{BO}_3 + \text{NaOH}$	120	129
10.0	$\text{H}_3\text{BO}_3 + \text{NaOH}$	360	380
10.5	$\text{H}_3\text{BO}_3 + \text{NaOH}$	1148	1174
11.0	NaOH	3587	3683

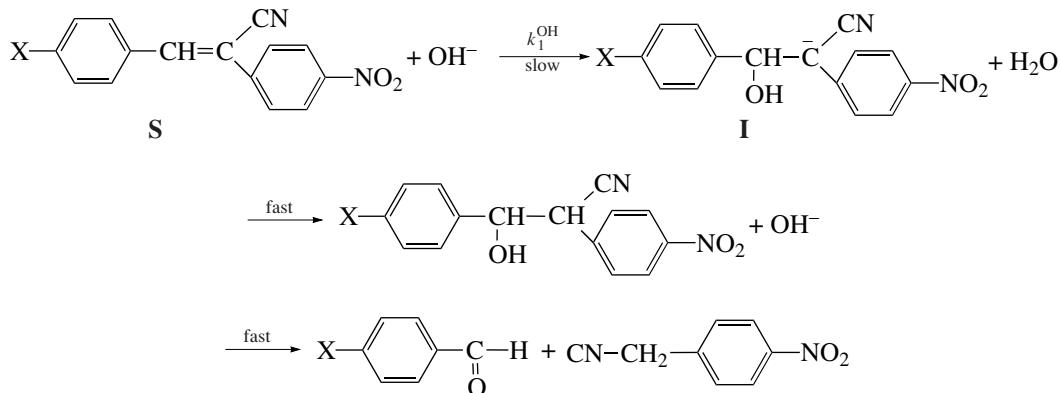
Note: $[\text{Substrate}] = 2.0 \times 10^{-5} \text{ M}$. The ionic strength of the buffer solutions was kept constant to 0.1 M by adding sodium chloride except at pH 1.0. Average rate constant for two or more kinetic runs. Estimated uncertainty, $\pm 3\%$.

Table 2. Rate constants for the hydrolysis of NCPN derivatives at various pH and 40°C

pH	$k_{\text{obs}}(\text{s}^{-1}) \times 10^6$			
	$p\text{-OCH}_3$	$p\text{-CH}_3$	$p\text{-Cl}$	$p\text{-NO}_2$
1.0	0.997	0.891	2.51	7.94
2.0	1.38	1.29	2.50	7.89
3.0	1.39	1.45	3.55	7.93
4.0	1.51	1.43	3.58	9.12
5.0	2.09	1.69	5.62	12.9
5.5	4.47	3.55	10.9	22.4
6.0	7.59	5.49	13.5	38.0
7.0	10.7	8.13	24.5	52.3
8.0	12.9	8.27	28.2	63.1
8.5	16.2	15.8	34.7	69.2
9.0	35.5	35.5	70.8	101
9.5	178	70.8	178	355
10.0	229	257	794	1071
10.5	1023	708	1549	3715
11.0	2691	2512	7079	8709

Note: $[\text{Substrate}] = 2.0 \times 10^{-5} \text{ M}$. Average rate constant for two or more kinetic runs. Estimated uncertainty, $\pm 3\%$.

At high pH, since the rate constant is directly proportional to hydroxide ion concentration, the following reaction mechanism is proposed (Scheme 1).



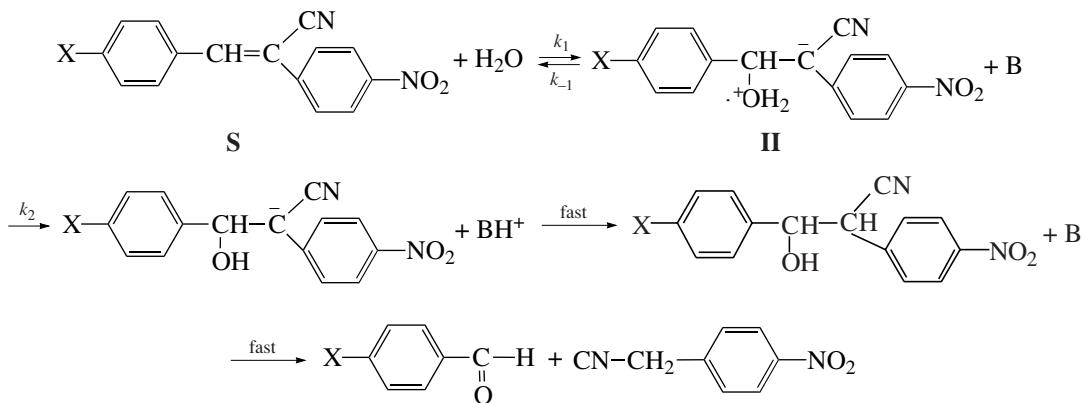
Scheme 1.

The α -carbon of carbanionic intermediate **I** is expected to be basic enough to abstract a proton from water under the reactions. The formation of an intermediate such as **I** has been previously proposed by Bernasconi [27] and Rappoport [28] et al. in the nucleophilic additions to olefins. Even though the intermediate **I** has not been observed directly in the nucleophilic additions, they have been detected under reaction conditions conducive to nucleophilic vinylic substitution [29, 30].

The rate-limiting step may be assessed by the Hammett ρ value. The magnitude of ρ , whether positive or negative, provides information about the rate-determin-

ing step of the reaction. A larger ρ indicates high sensitivity to substituent groups and implies that there is a large redistribution of charge involved in forming in the transition state [31]. Figure 3 shows that the Hammett ρ value 0.47 at pH 9.0 indicates a negative charge development at the reaction site in the transition state. Since the electron density at the reaction site increases in the first step and decreases in the second step, the positive ρ value would be consistent if the first step is rate limiting.

A plausible mechanism under acidic conditions is Scheme 2, which is similar to that shown in the basic condition except that water is added to [S].



Scheme 2.

Although the intermediate **II** could not be detected experimentally, Bernasconi et al. have previously proposed that the nucleophilic additions to activated olefins proceed through a zwitterionic intermediate **II** [32–35]. In the first step, either H_2O or OH^- may added to [S]. However, the second possibility has been ruled out because the hydroxide ion concentration is much smaller than that of water.

In the acidic media, the rate-determining step of the reaction may be deduced by the ρ value and general base effect. In the acidic solution, the third step is not rate limiting because the α -carbon of the carbanionic intermediate is expected to be basic enough to abstract a proton from hydronium. The ρ value measured at pH 3.0 is 0.80, indicating a negative charge development at the reaction site in the transition state. Since the addi-

tion of water and deprotonation from the intermediate **I** are expected to accelerate by the electron withdrawing group with the positive ρ value at pH 3.0, the rate determining step will be the first and second steps. On the other hand, the first step is expected to decrease but the deprotonation of the second is expected to increase by the electron withdrawing substituent at the β -aryl ring. Since the two effects are in opposition, the rate constant k_t should be insensitive to change in the basic solution.

However, the rate constant k_1^{OH} would be less sensitive to change in σ than k_1 because the hydroxide ion should be more reactive than water in the addition step ($\rho = 0.80$ at pH 3.0 and $\rho = 0.47$ at pH 9.0). Additional evidence in support of this conclusion is provided by the general base catalysis. Figure 4 shows that $\log k_{\text{bsd}}$ increases linearly with the acetate ion concentration when $[\text{AcO}^-] < 0.1$ M. This indicates that the general base catalyzes the deprotonation step and that k_2^{B} is involved in the rate-limiting step under these conditions. On the other hand, the rate of hydrolysis approaches the limiting values when $[\text{AcO}^-] > 0.1$ M under this condition, $k_2^{\text{B}}[\text{AcO}^-] \gg k_{-1}$ and k_1 becomes the rate-determining step.

The pH-rate profile shown in Fig. 2 provides convincing evidence in support of the proposed mechanism under all reaction conditions. The observed rate constants (k_t) can be divided into two parts, each representing a different mechanism

$$k_t = k_{11} + k_1^{\text{OH}}[\text{OH}^-], \quad (2)$$

where k_{11} is the first order rate constant for the spontaneous hydrolysis and k_1^{OH} is the catalytic rate constant for the base catalyzed reactions.

If the reaction proceeds as shown in Scheme 2, one can apply the steady-state approximation with respect to **III** [36, 37]. The rate equation can be expressed as Eq. (3)

$$\begin{aligned} \text{Rate} &= \frac{k_1 k_2 [\text{NPCN}]}{dt} = k_{11} [\text{NPCN}] \\ &= \frac{k_1 k_2 \sum [\text{B}]}{k_{-1} + k_2 \sum [\text{B}]} [\text{NPCN}], \end{aligned} \quad (3)$$

$$k_{11} = \frac{k_1 k_2 \sum [\text{B}]}{k_{11} + k_2 \sum [\text{B}]}, \quad (4)$$

$$\frac{1}{k_{11}} = \frac{k_{-1} + k_2 \sum [\text{B}]}{k_1 k_2 \sum [\text{B}]}, \quad (5)$$

$$\frac{1}{k_{11}} = \frac{1}{k_1} + \frac{1}{\left(\frac{k_1}{k_{-1}}\right) k_2 \sum [\text{B}]}. \quad (6)$$

From Eq. (6), it can be shown that if the concentration of the general base is high ($k_2[\text{B}] \gg k_{-1}$), then $k_{11} = k_1$. Therefore, if the concentration of the general base is high, k_{11} will take 1.34×10^{-5} s⁻¹ which is the limiting value of general base catalysis in Fig. 4. If water and the hydroxide ion are the only general bases present, Eq. (6) becomes

$$\frac{1}{k_{11}} = \frac{1}{k_1} + \frac{1}{\left(\frac{k_1}{k_{-1}}\right) \{k_2^{\text{H}_2\text{O}}[\text{H}_2\text{O}] + k_2^{\text{OH}}[\text{OH}^-]\}}. \quad (7)$$

Below pH 3.0, the concentration of hydroxide ions is negligible; then Eq. (7) becomes

$$\frac{1}{k_{11}} = \frac{1}{k_1} + \frac{1}{\left(\frac{k_1}{k_{-1}}\right) \{k_2^{\text{H}_2\text{O}}[\text{H}_2\text{O}]\}}. \quad (8)$$

The value of $(k_1/k_{-1})k_2^{\text{H}_2\text{O}}$ can be determined from the $k_1 = 1.34 \times 10^{-5}$ s⁻¹ and the observed rate constant $k_t = 1.82 \times 10^{-6}$ s⁻¹ at pH 2.0. The value is 2.11×10^{-6} s⁻¹. By substituting $k_t = 5.25 \times 10^{-6}$ s⁻¹ at pH 5.5 into Eq. (7), $(k_1/k_{-1})k_2^{\text{OH}} = 2.06 \times 10^3$ s⁻¹ can be obtained. As a result k_{11} becomes

$$\frac{1}{k_{11}} = \frac{1}{1.34 \times 10^{-5}} + \frac{1}{2.11 \times 10^{-6} + 2.06 \times 10^3 [\text{OH}^-]},$$

$$\frac{1}{k_{11}} = \frac{1.55 \times 10^{-5} + 2.06 \times 10^3 [\text{OH}^-]}{2.83 \times 10^{-11} + 2.76 \times 10^{-2} [\text{OH}^-]}, \quad (9)$$

$$k_{11} = \frac{2.83 \times 10^{-11} + 2.76 \times 10^{-2} [\text{OH}^-]}{1.55 \times 10^{-5} + 2.06 \times 10^3 [\text{OH}^-]}.$$

At pH 10.0, where the rate is directly proportional to the hydroxide ion concentration, k_t was found to be 3.67×10^{-4} s⁻¹. By substituting the above data into Eq. (2), $k_1^{\text{OH}} = 3.67$ s⁻¹ can be obtained.

As a result, the overall rate constant equation becomes

$$\begin{aligned} k_t &= \frac{2.83 \times 10^{-11} + 2.76 \times 10^{-2} [\text{OH}^-]}{1.55 \times 10^{-5} + 2.06 \times 10^3 [\text{OH}^-]} \\ &\quad + 3.67 [\text{OH}^-]. \end{aligned} \quad (10)$$

Table 1 and Fig. 2 show that the values of the overall rate constant, k_t , calculated by Eq. (10) are good agreement with observed values.

If an acetate ion is present as a general base catalyst, Eq. (6) becomes

$$\frac{1}{k_{11}} = \frac{1}{k_1} + \frac{1}{\left(\frac{k_1}{k_{-1}}\right)\{k_1^{\text{H}_2\text{O}}[\text{H}_2\text{O}] + k_2^{\text{OH}}[\text{OH}^-] + k_2^{\text{OAc}}[\text{OAc}^-]\}}. \quad (11)$$

From the values of $k_1 = 1.34 \times 10^{-5} \text{ s}^{-1}$, $(k_1/k_{-1})k_1^{\text{H}_2\text{O}}[\text{H}_2\text{O}] = 2.11 \times 10^{-6} \text{ s}^{-1}$, $(k_1/k_{-1})k_1^{\text{OH}} = 2.06 \times 10^3 \text{ s}^{-1}$, $[\text{OH}^-] = 6.03 \times 10^{-10} \text{ M}$, $[\text{CH}_3\text{COO}^-] = 0.1 \text{ M}$, and observed rate constant k_t , the value of the catalytic constant of the acetate ion $(k_1/k_{-1})k_2^{\text{OAc}} = 8.81 \times 10^{-3} \text{ s}^{-1}$ can be obtained. By substituting the above data into Eq. (11), k_{11} is given by Eq. (11-1).

$$\frac{1}{k_{11}} = \frac{1.68 \times 10^{-5} + 8.91 \times 10^{-3}[\text{OAc}^-]}{4.49 \times 10^{-11} + 1.18 \times 10^{-7}[\text{OAc}^-]}, \quad (11-1)$$

$$k_{11} = \frac{4.49 \times 10^{-11} + 1.18 \times 10^{-7}[\text{OAc}^-]}{1.68 \times 10^{-5} + 8.81 \times 10^{-3}[\text{OAc}^-]}. \quad (11-2)$$

As a result, the overall rate constant becomes

$$k_{11} = \frac{4.49 \times 10^{-11} + 1.18 \times 10^{-7}[\text{OAc}^-]}{1.68 \times 10^{-5} + 8.81 \times 10^{-3}[\text{OAc}^-]} + 3.67[\text{OH}^-]. \quad (11-3)$$

Figure 4 shows that the values of the overall rate constant, k_t , calculated by Eq. (11-3) are in good agreement with the observed values.

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