Cooxidation of Anilides and Oxalic Acid by Chromic Acid: A One-Step, Three-Electron Oxidation

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ABSTRACT: The chromic acid oxidation of a mixture of oxalic acid and anilides proceeds much faster than that of either of the two substrates alone. The oxidation kinetics of acetanilide, *p*-methyl-, *p*-chloro-, and *p*-nitroacetanilides by Cr(VI) in the presence of oxalic acid in aqueous acetic acid medium follows first-order, zero-order, and second-order dependence in [oxidant], [substrate], and in [oxalic acid], respectively, while the oxidation kinetics of benzanilide, *p*-methyl-, *p*-chloro-, and *p*-nitrobenzanilides follow first order in [oxidant] and fractional order each in [substrate] and [oxalic acid] and yields corresponding azobenzenes and benzaldehydes in the case of benzanilide and substituted benzanilides as the main products of oxidation. Aluminium ions suppress the reaction. The intermediate is believed to be formed from the anilide and a chromic acid-oxalic acid complex. In the proposed mechanism, the rate-limiting step involves the direct reduction of Cr(VI) to Cr(III). © 2000 John Wiley & Sons, Inc. Int J Chem Kinet 33: 21–28, 2001

INTRODUCTION

Enough evidences are available to show that the hexavalent chromium functions both as a one- and two-electron oxidant depending on the substrate. Rocek and Hasan [1] and Venkatasubramanian et al. [2] showed that the system involving an alcohol-oxalic acid-Cr(VI) behaved in a different way in that the Cr(VI) species get reduced directly to Cr(III) species involving three-electron transfer. Haight and Huber [3] proposed the rate law: Rate $= -d[\mathrm{Cr(VI)}]/dt = k'[\mathrm{HCrO_4^-}][\mathrm{Mn(C_2O_4)_2^{2-}}][\mathrm{C_2O_4^{2-}}]\,[\mathrm{H^+}]^2$ for the oxidation of manganese (II) by Cr(VI) in the presence of oxalate ion. It was observed that the rates of oxidation of piperidin–4-ols by Cr(VI) in the presence of oxalic acid were nearly ten times greater than the oxidation rates of piperidin–4-ols in the absence of oxalic acid [4]. While studying the influence of oxalic acid on Cr(VI) oxidation of As(III) in the presence of chloride ion in aqueous acetic acid, it was observed that both As(III) and oxalic acid are oxidized concertedly in three equivalent steps [5]. In their study of the kinetics and mechanism of oxidation of *o*-, *p*-, and *m*-substituted *trans*-cinnamic acids by pyridinium chlorochromate in the presence of oxalic acid, Mohan et al. [6] have proposed the carbanion formation for electron-withdrawing substituents and carbonium ion formation for electron-releasing substituents in their transition state. Their study revealed that both electron-releasing and electron-withdrawing substituents facilitate the rate of oxidation. The oxidation of phenoxy acetic acid by pyridinium fluorochromate in the presence of oxalic acid proceeds through pyridinium fluo-

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rochromate-oxalic acid complex, and the reaction involves three-electron transfer [7]. Substituted *s*-phenylmercaptoacetic acids were subjected to oxidative cleavage by chromic acid in the presence of oxalic acid, in which the formation of a ternary complex between oxalic acid, chromic acid, and *s*-phenylmercaptoacetic acid in a fast step is proposed [8]. The complex is then hydrolyzed in a subsequent slow rate-limiting step, yielding the corresponding sulphoxides. The title investigation is projected toward the behavior of the ternary system involving anilide-oxalic acid-Cr(VI) to evaluate the substituent effects, if any, in the cooxidation process, as anilides are very sluggish toward Cr(VI). Detailed literature scanning reveals that no satisfactory work is on record about the kinetics and mechanism of oxidation of anilide, substituted anilides, benzanilide, and substituted benzanilides by Cr(VI) in the presence of oxalic acid in aqueous acetic acid medium. Only a few reports are available about the oxidation kinetics of acetanilides and substituted acetanilides with a variety of oxidizing reagents [9–15].

EXPERIMENTAL

Materials

Acetanilide (AA) and benzanilide (BA) were LR grade and were used after recrystallization as described in the literature [16]. Substituted acetanilides and benzanilides like *para*-bromoacetanilide(*p*-BrA), *para*-chloroacetanilide (*p*-ClA), *para*-methylacetanilide (*p*-CH_3A), *para*-nitroacetanilide (*p*-NO_2A), *para*-chlorobenzanilide (*p*-ClBA), *para*-methylbenzanilide (*p*-CH_3BA), and *para*-nitrobenzanilide (*p*-NO_2BA) were prepared by the standard procedure, recrystallized as described in the literature, and their purities checked by determining their melting points, comparing with the values given in the literature [16,17] and by IR. All other reagents used were Analar grade. Conductivity water was used throughout the course of the investigation. Acetic acid (AR) was twice distilled over chromic oxide containing acetic anhydride (bp 118°C).

Kinetic Measurements

Standard solutions of anilides and Cr(VI) prepared in HOAc-H_2O mixtures were thermostatted for 2 h before each run. The reactions were carried out under pseudo-first-order conditions by keeping an excess of anilide over oxidant in the presence of oxalic acid, maintaining the ionic strength constant (0.2 M) by adding sodium perchlorate. All the reactions were carried out in 150 ml gena glass flasks coated black on the outside to avoid any photochemical decomposition. Aliquots (5 ml) of the reaction mixture were pipetted out at regular intervals and quenched in ice-cold 5% KI solution (5 ml) and 2 N H_2SO_4 (10 ml) was added. The reaction was monitored by estimating the unreacted Cr(VI) at various time intervals by iodometric procedure to a starch end point. Under the conditions of the experiment, the solvents were not oxidized. Rate constants were computed from the linear ($r > 0.98$) plot of log[Cr(VI)] against time. In the evaluation of rate coefficients, the kinetics were followed to nearly 75% reaction. Duplicate kinetic runs showed that the rates were reproducible within ±4%. The second-order rate constant, k_2, was obtained from the relation $k_2 = k_1/[\text{anilide}]$, where k_1 is the pseudo-first-order rate constant. The rate constants were calculated by the method of least squares.

Product Analysis

The product analyses were carried out separately for acetanilide and for benzanilides under kinetic conditions. Anilide (acetanilide 13.50 g, 0.1 M; benzanilide 19.70 g, 0.1 M), oxalic acid (12.60 g, 0.1 M), and Cr(VI) (1.0 g, 0.01 M) were made up to 50 ml with 45, 50, and 60% (v/v) acetic acid in different reaction bottles, keeping the ionic strength (0.2 M) constant.

To ensure the completion of the reaction, the reaction mixtures were allowed to stand for 12 h and were then diluted with water. The organic layers were extracted with ether and dried with anhydrous sodium sulphate. The solvents were removed under reduced pressure. The products of oxidation were cooled well and a small amount of cold dilute acetic acid was added to each sample. A sufficient amount of ice-cold bromine in acetic acid was added to each sample and shaken vigorously for half an hour after adding crushed ice. Bromo derivatives were separated out from each sample, filtered, dried, weighed, and recrystallized from ethanol and again dried and weighed. Comparison of the melting points of each bromo derivative with that of the authentic samples confirmed that one of the products of oxidation is azobenzene.

Sodium bicarbonate was added to each filtrate just to neutralize the solutions. After concentrating these solutions, ether extractions were carried out. After evaporating the ether, an excess of the concentrated solution of sodium bisulphite was added to each sample and shaken vigorously. The bisulphite addition compounds formed were filtered separately and treated with a slight excess of sodium carbonate solution. Oily layers separated out from each sample were extracted with ether. After evaporating the solvents, the samples in different reaction bottles of different composition were treated separately with an excess of freshly prepared saturated solution of 2,4-dinitrophenylhydrazine in 2 M hydrochloric acid. The precipitates formed were collected separately by filtration. The difference between the crude yield and yield determined after recrystallization was about 3–4%. The melting points of 2,4-dinitrophenylhydrazones (DNP) obtained from different samples of different composition, TLC of DNP of the products, and that of authentic samples and melting points confirmed that one of the products in the case of benzanilide was benzaldehyde. In the case of acetanilide, one of the products is acetaldehyde. The same procedure was adopted for benzanilides with different substituents. Under the specified conditions of oxidations of benzanilides by Cr(VI) in the presence of oxalic acid in aqueous acetic acid medium, it was observed that azobenzene is the only nitrogen-containing major product of oxidation along with benzaldehyde. In the case of substituted benzanilides containing substituents in the aniline moiety, substituted azobenzenes are the only nitrogen-containing major products of oxidation along with unsubstituted benzaldehydes. As there is no evidence for the presence of benzoic acids, it can be confirmed that the further oxidation of benzaldehydes did not take place. In the case of acetanilide, 2,4-dinitrophenylhydrazone was rather poor in yield.

RESULTS AND DISCUSSION

The oxidation of acetanilide, substituted acetanilides, benzanilide, and substituted benzanilides by Cr(VI) in the presence of oxalic acid in aqueous acetic acid medium showed the following features.

Rate Laws

1. Order with respect to oxidant: Under the condition [anilide] $\gg$ [Cr(VI)] the order in [Cr(VI)] is unity, as revealed by the linear plot of log[Cr(VI)] vs. time (Table I)
2. Order with respect to substrates: A more than threefold variation in the [acetanilides] has no effect on first-order rate constant (k_1) values, suggesting an independent nature of the rate in the [acetanilides] (Table I), whereas it follows fractional order in [BA] and [substituted BA], as revealed by the slopes of the plots of log k_1 against log[substrates] (Table I; Fig. 1).
3. Order with respect to oxalic acid: In the case of acetanilide, the order with respect to oxalic acid is found to be 1.95, whereas it is 1.28 in the case of BA, 1.39 in the case of p-CH_3BA, 1.72 in the case of p-ClBA, and 1.41 in the case of p-NO_2BA (Table I; Fig. 2)
4. Effect of varying ionic strength of the medium: The rate of oxidation of acetanilides as well as benzanilides remains constant when the ionic strength of the medium is increased.
5. Effect of adding sodium chloride: There is no change in the rates of oxidation of acetanilides and benzanilides by the addition of sodium chloride.
6. Effect of adding sulfuric acid and perchloric acid: The rates of oxidation of acetanilides as well as benzanilides are not at all affected by the addition of sulfuric acid and perchloric acid. They remain constant and do not vary.
7. Effect of adding pyridine: An appreciable decrease in the rate is observed by the addition of pyridine.
8. Effect of adding aluminium nitrate: Addition of aluminium nitrate suppresses the oxidation of benzanilide with Cr(VI) in presence of oxalic acid (Table II).
9. Effect of different substituents: It is observed that the substituents like -CH_3, -Cl, -NO_2 enhance the rate of oxidation of substituted BA by Cr(VI) in the presence of oxalic acid in aqueous acetic acid medium, and hence the observed trend in rate can be given as -CI $>$ -NO_2 $>$ -CH_3 $>$ -H (Table I). It is to be noted that Hammett relation could not be verified; even a more refined treatment as suggested by Venkatasubramanian et al. [2] could not lead to the evaluation of ρ.

The results of the title investigation study can be compared with the results obtained by Rocek and Hasan [1] and Venkatasubramanian et al. [2] except for the fact that the oxidation of acetanilides and benzanilides is very sluggish in the absence of oxalic acid. Radharkrishnamoorti and others [9–12,14] report the fractional order dependence on substrate in the case of oxidation kinetics study of acetanilides and substituted acetanilides with a variety of oxidizing agents in the absence of oxalic acid. It is observed that in the present study the rate increases with increasing oxalic acid concentration (Table I; Fig. 2). As suggested by Rocek and Hasan [1], this behavior is characteristic of the formation of an intermediate complex. Therefore, it is worthwhile to assume that chromic acid under the reaction conditions forms a complex with oxalic acid. This assumption is expressed in Eqs. (1) and (2), where C_1 is a 1:1 complex of oxalic acid and chromic acid.

Table I Effect of Varying [Reactants] on the Reaction Rate (I = 0.2 mol dm^{-3}; Temp: 308 K; Solvent: HOAc (50% v/v)).

10^2 [anilides] mol dm^{-3}	10^3 [Cr(VI)] mol dm^{-3}	10^2 [OxH$_2$] mol dm^{-3}	10^4 k_1 (S^{-1})								
			AA	*p*-ClA	*p*-BrA	*p*-NO$_2$A	*p*-CH$_3$A	BA	*p*-ClBA	*p*-NO$_2$BA	*p*-CH$_3$BA
0.6	1.0	4.0	4.40	4.35	4.27	4.15	4.23	3.95	16.87	7.49	4.57
1.0	1.0	4.0	4.28	4.18	4.37	3.98	4.19	4.48	19.00	8.56	5.22
1.2	1.0	4.0	4.39	4.23	4.54	4.21	4.25	4.69	19.95	8.96	5.37
1.6	1.0	4.0	4.52	4.16	4.37	4.26	4.29	5.13	21.40	9.55	5.89
2.0	1.0	4.0	4.35	4.29	4.25	4.31	4.51	5.32	22.51	10.17	6.24
2.4	1.0	4.0	4.42	4.31	4.36	4.23	4.44	5.60	23.40	10.66	6.53
							r	0.99	0.99	0.99	0.99
							Slope	0.25	0.24	0.25	0.26
1.0	0.4	4.0	4.34	4.15	4.52	4.02	4.12	4.52	18.85	8.64	5.19
1.0	1.2	4.0	4.25	4.24	4.18	4.11	4.41	4.39	19.25	8.48	5.40
1.0	1.6	4.0	4.19	4.22	4.25	4.21	4.32	4.42	19.11	8.58	5.32
1.0	2.0	4.0	4.27	4.30	4.31	4.01	4.16	4.51	18.92	8.51	5.27
1.7	1.5	1.0	0.29	—	—	—	—	0.92	1.70	1.40	1.05
1.7	1.5	2.0	1.12	—	—	—	—	2.00	5.24	3.67	2.39
1.7	1.5	3.0	2.45	—	—	—	—	3.47	10.71	6.53	3.77
1.7	1.5	4.0	4.31	—	—	—	—	5.15	20.25	9.77	6.07
1.7	1.5	5.0	6.76	—	—	—	—	7.08	26.91	13.96	9.55
1.7	1.5	6.0	9.55	—	—	—	—	9.12	34.48	17.56	12.90
—	—	*r*	0.99	—	—	—	—	0.99	0.99	0.99	0.99
		Slope	1.95	—	—	—	—	1.28	1.72	1.41	1.39

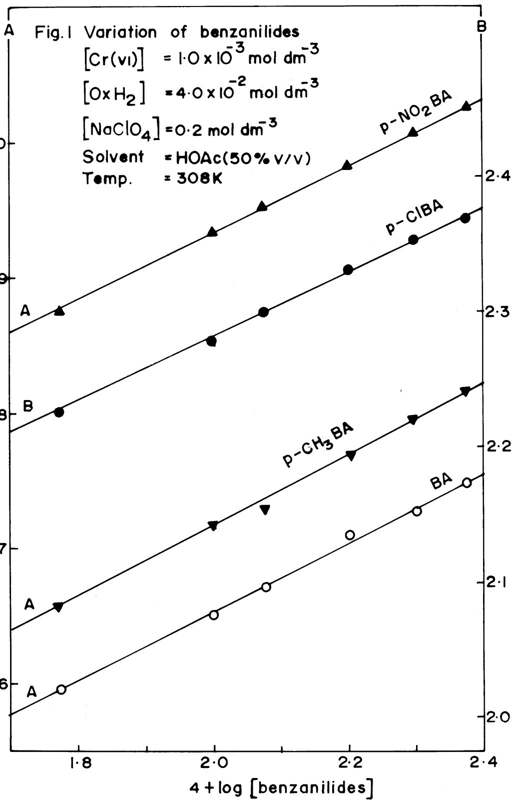


Figure 1 Variation of benzanilides.

$$(\mathrm{COOH})_2 + \mathrm{Cr(VI)} \underset{}{\overset{K_1}{\rightleftharpoons}} \mathrm{C}_1 \tag{1}$$

$$\mathrm{C}_1 + \mathrm{BA} \xrightarrow{k} \text{Products} \tag{2}$$

The concentration is given by Eq. (3)

$$[\mathrm{C}_1] = \frac{K_1[(\mathrm{COOH})_2][\mathrm{Cr(VI)}]_{\mathrm{Tot}}}{1 + K_1[(\mathrm{COOH})_2]} \tag{3}$$

Where

$$[\mathrm{Cr(VI)}]_{\mathrm{Tot}} = [\mathrm{Cr(VI)}] + [\mathrm{C}_1]$$

Table II Effect of Adding Aluminium Nitrate [*p*-ClBA] = 1.0 × 10^{-2} mol dm^{-3}; [Cr(VI)] = 1.0 × 10^{-3} mol dm^{-3}; [OxH$_2$] = 4.0 × 10^{-2} mol dm^{-3}; I = 0.2 mol dm^{-3}; Temp: 308 K; Solvent: HOAc (50% v/v).

[Aluminium Nitrate] M	10^4 k_1 (S^{-1})
0.00	19.00
1.00	0.77
1.50	0.46
2.00	0.31
2.50	0.22

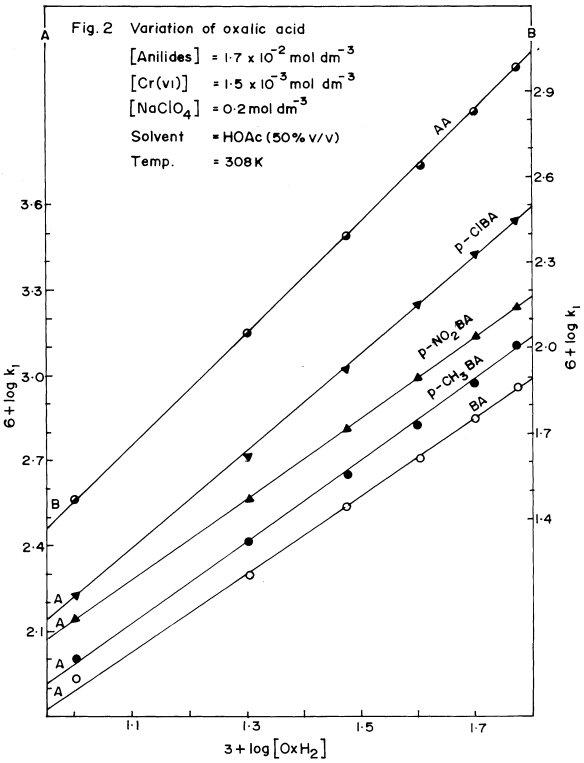


Figure 2 Variation of oxalic acid.

Since

$$V = k[C_1][BA]$$

$$V = k_{\text{exptl}}[\text{Cr(VI)}]_{\text{Tot}} \qquad (4)$$

$$[BA]/k_{\text{exptl}} = 1/k + 1/kK_1[(\text{COOH})_2] \qquad (5)$$

The validity of the Eq. (5) is tested by plotting $[BA]/k_{\text{exptl}}$ versus the reciprocal value of the oxalic acid concentration. A very good straight-line plot is obtained, which supports the assumption of the complex formation (Table I; Fig. 3).

The oxidation of the ternary system can be regarded as consisting of two independent reactions occurring simultaneously since the oxidation of anilides with Cr(VI) is very sluggish. Therefore, the following rate law can be given:

$$\begin{aligned}\frac{-d[\text{Cr(VI)}]}{dt} &= k_1[BA]^{0.25}[\text{OxH}_2]^{1.28}[\text{Cr(VI)}] \\ &\quad + k_2[\text{OxH}_2]^2[\text{Cr(VI)}] \\ &= k_{\text{obs}}[\text{Cr(VI)}]\end{aligned}$$

$$\begin{aligned}k_{\text{obs}} &= k_1\,[BA]^{0.25}[\text{OxH}_2]^{1.28} \\ &\quad + k_2[\text{OxH}_2]^2 \qquad (6)\end{aligned}$$

The first term represents the cooxidation process and is responsible for the observed rate increase of anilides oxidation by Cr(VI) in the presence of oxalic acid. At lower concentrations, the second term of the Eq. (6) can be neglected.

$$k_{\text{obs}} = k_1[BA]^{0.25}[\text{OxH}_2]^{1.28}$$

$$\frac{k_{\text{obs}}}{[BA]^{0.25}} = k_1[\text{OxH}_2]^{1.28} \qquad (7)$$

According to Eq. (7), a linear relationship should be obtained between $k_{\text{obs}}/[BA]$ and $[\text{OxH}_2]$ and between $k_{\text{obs}}/[\text{OxH}_2]$ and [BA]. In practice, straight-line graphs are obtained, but k values obtained from the slopes of these straight lines could not fit in Hammett equation.

If we consider the scheme

$$\text{OxH}_2 + \text{H}_2\text{CrO}_4 \overset{K_1}{\rightleftharpoons} \text{C}_1$$

$$\text{C}_1 + \text{BA} \overset{K_2}{\rightleftharpoons} \text{C}_2$$

$$\text{C}_2 \overset{k_3}{\rightleftharpoons} \text{Products}$$

$$\text{Rate} = k_3\,[\text{C}_2] = k_{\text{obs}}\,[\text{Cr(VI)}]$$

$$\text{C}_1 = K_1[\text{OxH}_2][\text{H}_2\text{CrO}_4]$$

$$\text{C}_2 = K_2[\text{C}_1][\text{BA}]$$

$$\begin{aligned}[\text{Cr(VI)}]_T = [\text{H}_2\text{CrO}_4] &+ K_1[\text{OxH}_2][\text{H}_2\text{CrO}_4] \\ &+ K_2K_1[\text{OxH}_2][\text{H}_2\text{CrO}_4][\text{BA}]\end{aligned}$$

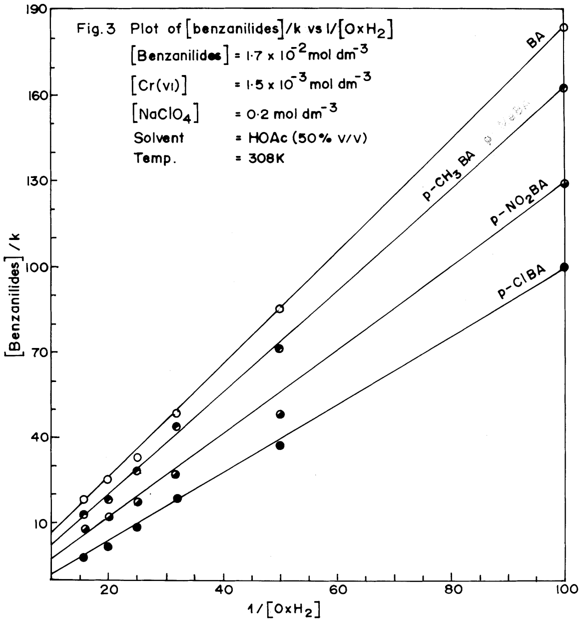


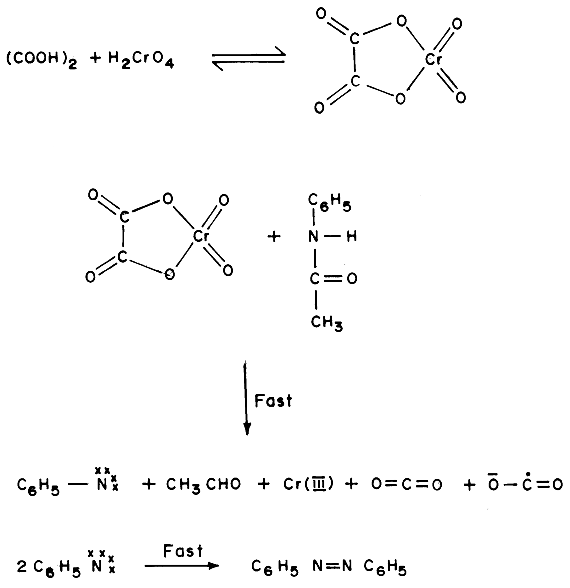
Figure 3 Plot of [benzanilides]/k vs 1/[OxH_2].

Rearranging, we get

$$\begin{aligned} k_3[C_2] &= k_{\text{obs}}[H_2CrO_4]\ \{1 + K_1[OxH_2] \\ &\quad + K_2K_1[OxH_2][BA]\} \\ &= k_3K_2K_1[OxH_2][H_2CrO_4][BA] \end{aligned}$$

$$\frac{[OxH_2]}{k_{\text{obs}}} = \left(\frac{1}{k_3K_2K_1} + \frac{[OxH_2]}{K_2K_1}\right)\left(\frac{1}{[BA]} + \frac{[OxH_2]}{k_3}\right)$$

This equation is similar to the equation obtained by Venkatasubramanian et al [2]. Therefore, $[OxH_2]$ remaining constant, a linear relationship should exist between $[OxH_2]/k_{\text{obs}}$ and $1/[BA]$, the intercept being $[OxH_2]/k_3$ (Fig. 4). But even in this case, Hammett relationship could not be verified. Considering all the kinetic factors, Scheme I is proposed for the oxidation of acetanilides by chromic acid in the presence of oxalic acid, which explains the independent nature of rate in the [acetanilides]. Scheme II and Scheme III are proposed for the oxidation of benzanilides by chromic acid in presence of oxalic acid. Both the schemes involve the formation of ternary complexes (oxalic acid–chromic acid–benzanilides). They undergo concerted intramolecular electronic rearrangement to yield the products in one step. Scheme III is preferred over Scheme II since it adequately explains the effect of sulfuric acid and perchloric acid on the rate of oxidation. Scheme II involves the shift of hydride ion, while Scheme III involves the shift of hydrogen ion. But in the kinetic results that the addition of sulfuric acid or perchloric acid bring out, no change in the rate constant values supports Scheme III, since the shift of hydrogen ion is hindered due to the addition of sulfuric acid and perchloric acid.



Scheme I

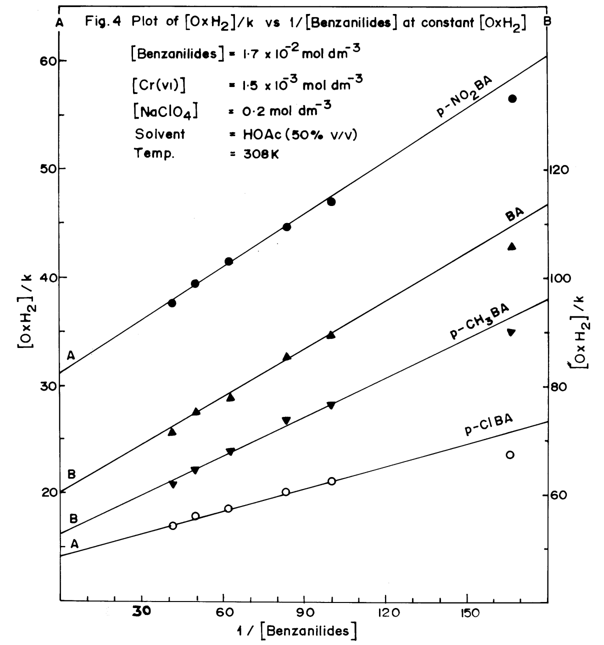
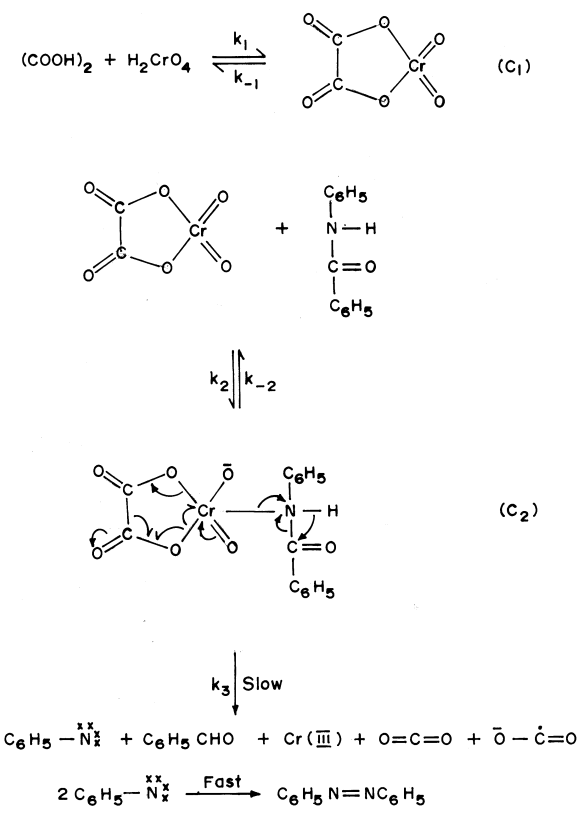
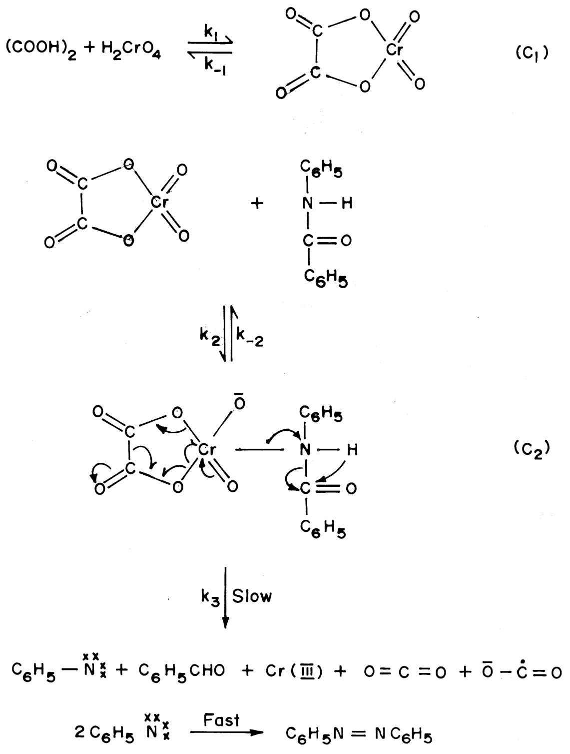


Figure 4 Plot of $[OxH_2/k$ vs. 1/[benzanilides] at constant $[OxH_2]$.



Scheme II



Scheme III

Table III Activation Parameters [Anilides] = 1.0 × 10^{-2} mol dm^{-3}; [Cr(VI)] = 1.0 × 10^{-3} mol dm^{-3}; [OxH_2] = 4.0 × 10^{-2} mol dm^{-3}; I = 0.2 mol dm^{-3}; Solvent; HOAc (50% v/v).

Anilides	$\Delta H\ddagger$ (kJ mol^{-1})	$-\Delta S\ddagger$ (J deg^{-1} mol^{-1})	$\Delta G\ddagger$ (kJ mol^{-1})
AA	44.62	125.36	84.62
p-CIA	47.36	118.95	84.00
p-CH_3A	44.31	128.90	84.01
p-NO_2A	45.57	118.62	84.25
BA	37.76	150.16	84.01
p-CH_3BA	37.60	149.41	83.61
p-CIBA	19.65	188.70	77.77
p-NO_2BA	29.87	170.20	82.30

The fact that the methyl, chloro, and nitro groups enhance the rate of oxidation of benzanilides and that the enhancement is more pronounced due to the presence of chloro and nitro groups supports the schemes. Addition of aluminium nitrate suppresses the oxidation of benzanilides with Cr(VI) in the presence of oxalic acid (Table II). Chatterjee and coworkers [18] have reported that the chromic acid oxidation of oxalic acid can be effectively prevented by the addition of aluminum nitrate. This effect is probably due to the ability to complex rather firmly with oxalic acid, and thus the formation of the oxalic acid–chromic acid complex, which is an intermediate in the oxidation reaction, is hindered. Rocek and Hasan [1] also report that the rate-accelerating effect of oxalic acid is almost canceled by the addition of aluminum nitrate. These results are in good agreement with the conclusion that the chromic acid–oxalic acid complex is an intermediate in the cooxidation reaction. The unusual high reactivity of the complex composed of a molecule of chromic acid and oxalic acid and a molecule of substrate is explained in this way [1]: that a complex containing both components offers the reaction a more favorable pathway than the oxidation of a single molecule. The presence of the molecule of oxalic acid within the complex facilitates the reduction of Cr(VI) directly to Cr(III), a direct three-electron reduction coupled with the formation of a very stable molecule of carbon dioxide and •COOH species, thus avoiding the formation of the energetically unfavorable Cr(IV) [1]. The fate of $^\bullet CO_2^-$ is explained as

$$^\bullet CO_2^- + \mathrm{Cr(VI)} \xrightarrow{\text{Fast}} \mathrm{Cr(V)} + CO_2$$

$$\mathrm{Cr(V)} + (\mathrm{COOH})_2 \xrightarrow{\text{Fast}} 2CO_2 + \mathrm{Cr(III)}$$

Earlier studies lend strong support for this scheme [20]. The activation enthalpies and entropies of the

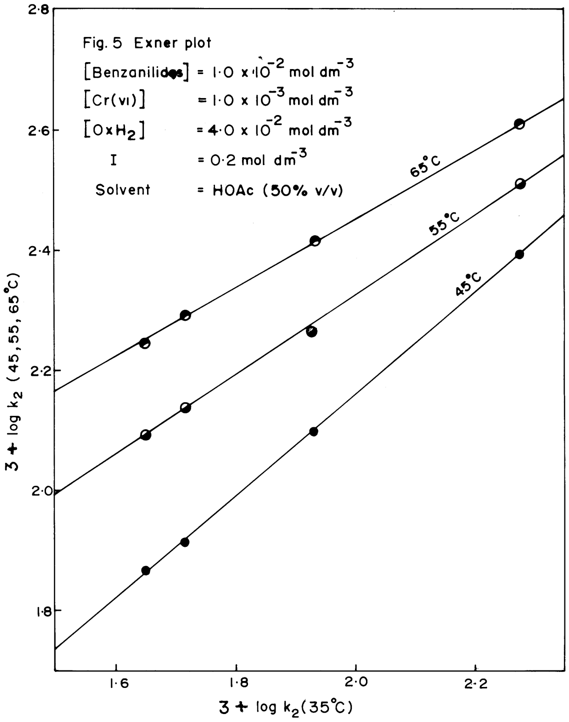


Figure 5 Exner plot.

oxidation of benzanilides by Cr(VI) in the presence of oxalic acid in aqueous acetic acid medium are linearly related (Table III). The correlation is tested and found genuine by applying Exner's criterion [19] (Fig. 5). The isokinetic temperature computed from these plots is 454 K. However, the values of activation enthalpies and values of free energy of activation of acetanilides remain constant.

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