Kinetics and Mechanism of the Oxidation of Aliphatic Alcohols by Quinolinium Fluorochromate

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ABSTRACT: The oxidation of some aliphatic alcohols by quinolinium fluorochromate (QFC) in dimethyl sulfoxide leads to the formation of corresponding carbonyl compounds. The reaction is first order with respect to QFC. The reaction exhibited Michaelis-Menten type kinetics with respect to the alcohol. The reaction is catalyzed by hydrogen ions. The hydrogen-ion dependence has the form: $k_{obs} = a + b[H^+]$. The oxidation of [1,1-$^2H_2$]ethanol ($MeCD_2OH$) exhibits a substantial primary kinetic isotope effect. The reaction has been studied in nineteen different organic solvents. The solvent effect was analyzed using Taft's and Swain's multiparametric equations. The rate of disproportionation of the complex is susceptible to both polar and steric effects of the substituents. A suitable mechanism has been proposed. © 1999 John Wiley & Sons, Inc. Int J Chem Kinet 31: 469–475, 1999

INTRODUCTION

Quinolinium fluorochromate (QFC) has been used as a mild and selective oxidizing reagent in synthetic organic chemistry [1]. However, there does not seem to be many reports on the kinetic and mechanistic aspects of oxidation reactions of QFC [2]. The kinetics of oxidation of alcohols by QFC has not been reported. We have been interested in the kinetics of reactions of complexed Cr(VI) species and have reported kinetics and mechanism of the oxidation of aliphatic alcohols by pyridinium fluorochromate (PFC) [3], pyridinium chlorochromate (PCC) [4] and 2,2'-bipyridinium chlorochromate(BPCC) [5]. It was observed that the oxidations by PCC and PFC presented different kinetic pictures. In this paper we describe the kinetics and mechanism of the oxidation of nine primary and five secondary aliphatic alcohols by QFC in dimethyl sulfoxide (DMSO) as solvent.

EXPERIMENTAL

Materials

The QFC was prepared by the reported method [1] and its purity was checked by an iodometric method. The procedure used for the purification of alcohols have been described earlier [6]. [1,1-$^2H_2$]Ethanol ($MeCD_2OH$) was prepared by Kalpan's method [7]. Its isotopic purity, as ascertained by its NMR spectra, was 93 ± 5%. Due to the non-aqueous nature of the medium, p-toluenesulphonic acid (TsOH) was used as a source of hydrogen ions. TsOH is a strong acid and in a polar medium like DMSO it is likely to be completely ionised. Solvents were purified by the usual method [8].

Product Analysis

The product analysis were carried out under kinetic conditions. In a typical experiment, ethanol (2.30 g,

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0.05 mol) and QFC (2.49 g, 0.01 mol) were made up to 50 cm^3 in DMSO and kept in dark for about 15 hours to ensure the completion of the reaction. The solution was then treated with an excess (200 cm^3) of a saturated solution of 2,4-dinitophenylhydrazine in 2 mol dm^{-3} HCl and kept overnight in a refrigerator. The precipitated 2,4-dinitrophenylhydrazone (DNP) was filtered off, dried, weighed, recrystalized from ethanol and weighed again. The yield of DNP before and after recrystallization was 2.0 g (90%) and 1.7 g (75%), respectively. The DNP was found identical (m.p. and mixed m.p.) with the DNP of acetaldehyde. Similar experiments with other alcohols led to the formation of DNP of the corresponding carbonyl compounds in yields ranging from 71 to 83%, after recrystallization. Iodometric determinations of the oxidation state of chromium in completely reduced reaction mixtures indicated that the oxidation state of the reduced chromium species was 3.92 ± 0.21.

Spectral Studies

UV-VIS spectra (200–800 nm) of (i) QFC (0.001 mol dm^{-1}) alone and (ii) QFC (0.001 mol dm^{-1}) + 2-propanol (0.01 mol dm^{-1}), in DMSO as the solvent, were obtained on a HP-diode array fast scanning spectrophometer (Model 8452A) at room temperature ($\approx$300 K). The blank was DMSO. The scanning speed was 600 nm s^{-1}. The time elapsed between the mixing of the two reagents and recording of the spectrum (ii) was $>$20 s.

Kinetic Measurements

The reactions were followed under pseudo-first-order conditions by keeping a large excess (×15 or greater) of the alcohol over QFC. The temperature was kept constant to ±0.1 K. The solvent was DMSO, unless specified otherwise. The reactions were followed by monitoring the decrease in the concentration of QFC spectrophotometrically at 356 nm for 80% of the reaction. The pseudo-first-order rate constants, k_{obs}, were evaluated from the linear ($r = 0.990$–0.999) plots of log [QFC] against time. Duplicate kinetic runs showed that the rate constants were reproducible to within ±3%. Multivariate and simple linear regression analyses were carried out by the least-squares method.

RESULTS

The oxidation of alcohols results in the formation of corresponding carbonyl compounds. The overall reaction may be represented as equation (1).

$$R_2CHOH + CrO_2FO^-QH^+ \longrightarrow R_2CO + H_2O + CrOFO^-OH^+ \quad (1)$$

Thus QFC undergoes a two-electron change. This accords with the earlier observations with PFC [9] and QFC [2].

Rate Law

The reaction is first order with respect to QFC. Michaelis-Menten type kinetics were observed with respect to alcohols (Table I). A plot of 1/k_{obs} vs 1/[alcohol] is linear with an intercept on the rate ordinate. This indicates the following overall mechanism equations (2) and (3) and the rate law equation (4).

$$\text{Alcohol} + \text{QFC} \overset{K}{\rightleftharpoons} [\text{Complex}] \quad (2)$$

$$[\text{Complex}] \xrightarrow{k_2} \text{Product} \quad (3)$$

$$\text{Rate} = k_2\,K\,[\text{Alcohol}]\,[\text{QFC}]/(1 + K\,[\text{Alcohol}]) \quad (4)$$

The variation in alcohol concentration was studied at four temperatures and the values of k_2 and K were evaluated from the double reciprocal plots. The thermodynamic parameters for the complex formation and the activation parameters for its disproportionation were also calculated (Tables II and III).

Table I Rate Constants for the Oxidation of Ethanol by QFC at 298 K

10^3 [QFC] (mol dm^{-3})	[Ethanol] (mol dm^{-3})	[TsOH] (mol dm^{-3})	10^4 k_{obs} (s^{-1})
1.0	0.02	0.0	10.9
1.0	0.04	0.0	20.4
1.0	0.08	0.0	35.9
1.0	0.20	0.0	66.6
1.0	0.40	0.0	93.0
1.0	0.80	0.0	116
1.0	1.00	0.0	122
2.0	0.02	0.0	11.4
4.0	0.02	0.0	10.3
6.0	0.02	0.0	10.0
8.0	0.02	0.0	11.8
1.0	0.02	0.0	11.5*
1.0	0.04	0.1	23.1
1.0	0.04	0.3	28.2
1.0	0.04	0.5	34.4
1.0	0.04	0.8	43.2
1.0	0.04	1.0	50.1

* contained 0.001 mol dm^{-3} acrylonitrile

Table II Formation Constants and Thermodynamic Parameters of Alcohol-QFC Complexes in DMSO

		K/dm^3 mol^{-1}				Δ H	Δ S
S. No.	Alcohol	288 K	298 K	308 K	318 K	(kJ mol^{-1})	(J mol^{-1}K^{-1})
1	Methanol	3.52	2.85	2.33	1.68	−21.2 ± 0.8	−55 ± 3
2	Ethanol	4.51	3.81	3.30	2.65	−15.7 ± 0.7	−34 ± 2
3	Propanol-1	5.73	4.91	4.11	3.20	−17.1 ± 0.9	−36 ± 3
4	Butanol-1	4.10	3.31	2.69	2.00	−20.5 ± 0.9	−51 ± 3
5	Pentanol-1	4.75	3.90	3.21	2.45	−19.1 ± 0.8	−45 ± 3
6	2-Methyl-propanol-1	5.15	4.45	3.81	3.10	−15.2 ± 0.7	−31 ± 2
7	2-Chloro-ethanol	4.80	4.15	3.41	2.71	−17.0 ± 0.9	−38 ± 3
8	2-Methoxy-ethanol	3.95	3.15	2.50	1.91	−20.7 ± 0.8	−52 ± 3
9	2,2-Dimethyl-propanol-1	4.85	4.21	3.53	2.85	−15.9 ± 0.8	−34 ± 2
10	Propanol-2	5.32	4.50	3.80	2.83	−18.1 ± 1.0	−41 ± 3
11	Butanol-2	3.91	3.35	2.50	1.82	−22.1 ± 1.1	−57 ± 4
12	Pentanol-2	4.39	3.63	2.86	2.08	−21.3 ± 1.0	−53 ± 3
13	1-Chloro-propanol-2	5.10	4.50	3.73	3.03	−15.8 ± 0.9	−33 ± 2
14	1-Methoxy-propanol-2	4.50	3.63	2.89	2.40	−18.6 ± 0.8	−44 ± 2
15	MeCD$_2$OH	4.50	3.61	2.90	2.11	−21.9 ± 0.6	−55 ± 2

Induced Polymerization of Acrylonitrile

The oxidation of alcohols, in an atmosphere of nitrogen, failed to induce polymerization of acrylonitrile. Further, the addition of acrylonitrile did not affect the rate. This indicates that a one-electron oxidation, giving rise to free radicals, is unlikely in the present reaction (Table I).

Kinetic Isotope Effect

To ascertain the importance of cleavage of the C—H bond in the rate-determining step, oxidation of [1,1-$^2H_2$]ethanol was studied. The results showed the presence of a substantial primary kinetic isotope effect (Table III).

Spectral Studies

A perusal of the spectra of QFC and QFC + 2-propanol showed that there is a distinct change in the absorption spectra of QFC on the addition of 2-propanol (Figure 1). It was further observed that the absorbance due to the intermediate decreases with time. This supports the postulation of the formation of

Table III Rate Constants of Disproportionation and Activation Parameters of the Alcohol-QFC Complexes in DMSO

	10^4 k_2/s^{-1}				Δ H*	Δ S*	Δ G*
Alcohol[a]	288 K	298 K	308 K	318 K	(kJ mol^{-1})	(J mol^{-1}K^{-1})	(kJ mol^{-1})
1	0.95	2.65	7.68	21.8	78.1 ± 0.9	−52 ± 3	93.3 ± 0.7
2	71.0	154	345	748	57.4 ± 0.8	−87 ± 3	83.3 ± 0.7
3	116	256	565	1190	56.7 ± 0.5	−86 ± 2	82.1 ± 0.4
4	220	452	940	1950	52.8 ± 0.8	−94 ± 3	80.6 ± 0.7
5	248	510	1020	2130	51.8 ± 0.9	−96 ± 3	80.4 ± 0.7
6	370	765	1440	2980	49.9 ± 0.9	−99 ± 3	79.4 ± 0.8
7	1.40	3.8	11.2	27.5	73.7 ± 0.9	−63 ± 3	92.4 ± 0.8
8	11.8	28.2	71.5	160	64.1 ± 0.8	−79 ± 2	87.5 ± 0.6
9	3750	7660	14600	27400	47.8 ± 0.2	−87 ± 1	73.7 ± 0.1
10	4400	8890	17100	32000	47.8 ± 1.1	−86 ± 3	73.3 ± 1.0
11	7100	14000	26700	49200	46.6 ± 1.0	−86 ± 3	72.1 ± 1.0
12	11800	25000	48900	93000	49.8 ± 1.2	−71 ± 3	70.8 ± 1.4
13	156	382	932	2150	64.2 ± 0.7	−57 ± 2	81.1 ± 0.9
14	925	2100	4610	9500	56.7 ± 1.0	−68 ± 3	76.9 ± 1.1
15	12.9	30.1	71.5	164	62.1 ± 0.8	−86 ± 3	87.5 ± 0.9
k_H/k_D	5.50	5.12	4.83	4.56			

[a] For the identity of compounds see Table II

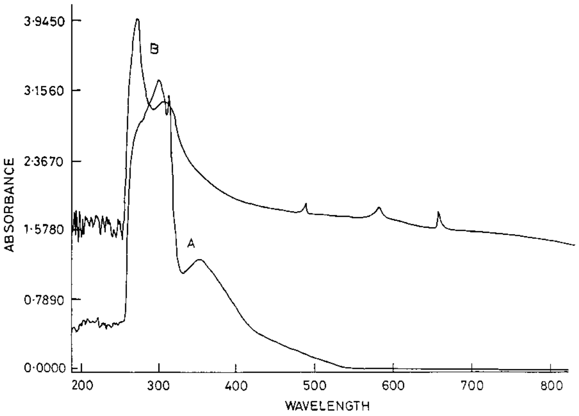


Figure 1 UV-VIS spectra of (A) 0.001 mol dm^{-3} QFC and (B) 0.001 mol dm^{-3} QFC + 0.10 mol dm^{-3} 2-propanol. Solvent : DMSO

an intermediate in a rapid pre-equilibrium and its subsequent decomposition to yield the ultimate product.

Effect of Acidity

The reaction is catalyzed by hydrogen ions. The hydrogen-ion dependence has the following form equation (5). The results are recorded in Table I.

$$k_{obs} = a + b\,[H^+] \qquad (5)$$

To determine whether the acidity is affecting the formation constant, K, and/or the rate constant, k_2, of the disproportionation, the effect of variation in the alcohol concentration was studied at different acidities. Results showed that K is not appreciably affected by changes in the acidity whereas the value of k_2 vary. The values of a and b, for ethanol, are $1.96 \pm 0.1 \times 10^{-3}$ s^{-1} and $3.00 \pm 0.1 \times 10^{-3}$ mol^{-1} dm^3 s^{-1} respectively ($r^2 = 0.9986$).

Solvent Effect

The oxidation of ethanol was studied in 19 different organic solvents. The choice of solvent was limited due to the solubility of QFC and its reaction with primary and secondary alcohols. There was no reaction with the solvents chosen. The kinetics were similar in all the solvents. The values of K and k_2 are recorded in Table IV.

DISCUSSION

There is no significant isokinetic relationship between the activation enthalpies and entropies of the oxidation of fourteen alcohols ($r^2 = 0.5112$). A correlation between the calculated values of enthalpies and entropies of activation is often vitiated by the experimental errors associated with them. Exner [10] has suggested an alternative method of testing the validity of isokinetic relationship. An Exner's plot between log k_2 at 288 K and at 318 K was linear ($r^2 = 0.9970$). The value of isokinetic temperature evaluated from the Exner's plot is 1510 ± 62K. The linear isokinetic correlation implies that all the alcohols are oxidized by the same mechanism and are governed by the changes in both the enthalpy and entropy of the activation.

Solvent Effect

The rate constants, k_2, for the disproportionation of the complex, in eighteen solvents (CS_2 was not considered, as the complete range of solvent parameters was not available) were correlated in terms of the linear solvation energy relationship equation (7) of Kamlet et al. [12].

$$\log k_2 = A_0 + p\pi^* + b\beta + a\alpha \qquad (7)$$

Table IV Formation Constants and Rate Constants for the Decomposition of Ethanol-QFC Complexes in Different Solvents at 298 K

	K	$10^3\,k_2$		K	$10^3\,k_2$
Solvents	dm mol^{-1}	s^{-1}	Solvents	dm mol^{-1}	s^{-1}
Chloroform	6.21	18.6	Acetic acid	7.10	3.02
1,2-Dichloromethane	5.76	22.9	Cyclohexane	7.12	0.59
Dichloromethane	4.90	20.4	Toluene	7.63	5.01
DMSO	4.51	71.0	Acetophenone	4.80	28.2
Acetone	7.35	19.1	THF	6.57	9.33
N,N-Dimethylformamide	7.14	37.2	t-butyl alcohol	8.00	7.24
Butanone	6.45	7.24	1,4-Dioxane	7.21	10.2
Nitrobenzene	5.86	24.5	1,2-Dimethoxyethane	6.55	4.79
Benzene	8.11	6.92	Carbon disulfide	5.85	2.45
Ethyl acetate	4.90	7.59			

In this equation, π^* represents the solvent polarity, β the hydrogen bond acceptor basicities, and α is the hydrogen bond donor acidity. A_0 is the intercept term. It may be mentioned here that out of the 18 solvents, 13 have a value of zero for α. The results of correlation analyses in terms of eq. (7), a biparametric equation involving π^* and β, and separately with π^* and β are given below in equations. (8)–(11).

$$\begin{aligned}\log k_2 \\ = -4.12 + 1.71\ &\pi^* + \ 0.21\ \beta \ + \ 0.16\alpha \\ &(\pm 0.22) \quad (\pm 0.18) \quad (\pm 0.17)\end{aligned} \tag{8}$$

$$R^2 = 0.8527;\ \text{sd} = 0.20;\ n = 18;\ \gamma = 0.30$$

$$\begin{aligned}\log k_2 = -4.16 + 1.77\ (\pm 0.21)\ \pi^* \\ + 0.15\ (\pm 0.17)\ \beta\end{aligned} \tag{9}$$

$$R^2 = 0.8431;\ \text{sd} = 0.19;\ n = 18;\ \gamma = 0.30$$

$$\log k_2 = -4.13 + 1.81\ (\pm 0.20)\ \pi^* \tag{10}$$

$$r^2 = 0.8348;\ \text{sd} = 0.20;\ n = 18;\ \gamma = 0.30$$

$$\log k_2 = -3.13 + 0.47\ (\pm 0.39)\ \beta \tag{11}$$

$$r^2 = 0.829;\ \text{sd} = 0.47;\ n = 18;\ \gamma = 0.87$$

Here n is the number of data points and γ is the Exner's statistical parameter [13].

Kamlet's [12] triparametric equation explains about 85% of the effect of solvent on the oxidation. However, by Exner's criterion [13] the correlation is not even satisfactory (cf. eq. 8). The major contribution is of solvent polarity. It alone accounted for about 83% of the data. Both β and α play relatively minor roles.

The data on the solvent effect were analyzed in terms of Swain's equation [14] of cation- and anion-solvating concept of the solvents as well as equation (12).

$$\log k_2 = \text{aA} + \text{bB} + \text{C} \tag{12}$$

Here A represents the anion-solvating power of the solvent and B the cation-solvating power. C is the intercept term. (A + B) is postulated to represent the solvent polarity. The rates in different solvents were analyzed in terms of equation (12), separately with A and B and with (A + B).

$$\begin{aligned}&\log k_2 \\ &\quad = 0.65(\pm 0.02)\ \text{A} + 1.85\ (\pm 0.01)\ \text{B} - 4.36\end{aligned} \tag{13}$$

$$R^2 = 0.9990;\ \text{sd} = 0.02;\ n = 19;\ \gamma = 0.02$$

$$\log k_2 = 0.39(\pm 0.61)\ \text{A} - 3.10 \tag{14}$$

$$r^2 = 0.0236;\ \text{sd} = 0.49;\ n = 19;\ \gamma = 0.95$$

$$\log k_2 = 1.79(\pm 0.12)\ \text{B} - 4.15 \tag{15}$$

$$r^2 = 0.9336;\ \text{sd} = 0.13;\ n = 19;\ \gamma = 0.19$$

$$\log k_2 = 1.45 \pm 0.15\ (\text{A} + \text{B}) - 4.33 \tag{16}$$

$$r^2 = 0.8582;\ \text{sd} = 0.17;\ n = 19;\ \gamma = 0.28$$

The rates of disproportionation of the complex in different solvents showed an excellent correlation in Swain's equation with both cation and anion-solvating powers contributing toward the observed effect of the solvents. However, the contribution of the cation-solvation is relatively greater. A correlation with B alone showed that the cation-solvation accounts for about 93% of the data. The solvent polarity, represented by (A + B), accounted for about 86% of the data. In view of the fact that solvent polarity is able to account for about 86% of the data, an attempt was made to correlate the rate with the relative permittivity of the solvent. It was observed that a plot of log (rate) against the inverse of the relative permittivity is not linear ($r^2 = 0.5150$; sd = 0.35; $\gamma = 0.55$).

Correlation Analysis of Reactivity

The rates of oxidation of the alcohols failed to yield any significant correlation separately with Taft's σ^* and Es values equations (17) and (18)].

$$\log k_2 = -2.13(\pm 0.34)\ \Sigma\ \sigma^*\ 0.09 \tag{17}$$

$$r^2 = 0.7669;\ \text{sd} = 0.53;\ \gamma = 0.37;\ n = 14$$

$$\log k_2 = -1.22(\pm 0.38)\ \Sigma\ \text{E}_\text{s} - 0.25 \tag{18}$$

$$r^2 = 0.7111;\ \text{sd} = 0.66;\ \gamma = 0.41;\ n = 14$$

The rates were, therefore, correlated in terms of Pavelich-Taft's [15] dual substituent-parameter equation (19).

$$\log k_2 = {}^*\ \Sigma\ \sigma^* + \delta\ \Sigma\ \text{Es} + \log \text{k}_0 \tag{19}$$

Table V Temperature Dependence of the Reaction Constants

Temp./K	ρ^*	δ	R^2	sd	γ
288	-1.68 ± 0.04	-0.82 ± 0.02	0.9983	0.06	0.03
298	-1.60 ± 0.03	-0.81 ± 0.02	0.9984	0.05	0.03
308	-1.49 ± 0.04	-0.79 ± 0.02	0.9981	0.06	0.03
318	-1.42 ± 0.05	-0.76 ± 0.03	0.9966	0.07	0.04

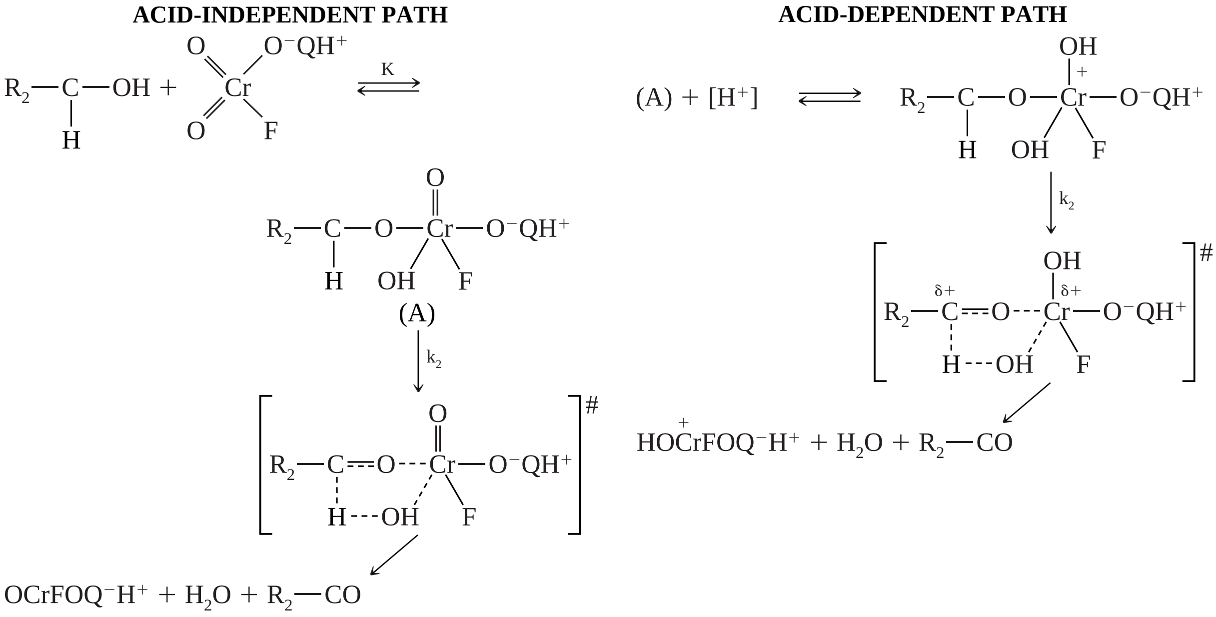
The values of substituent constants were obtained from the compilation by Wiberg [16]. The correlations are excellent: the reaction constants being negative (Table V). There is no significant collinearity ($r^2 = 0.2322$) between σ^* and Es values of the fourteen substituents.

The negative polar reaction constant indicates an electron-deficient carbon center in the transition state of the rate-determining step. The negative steric reaction constant shows a steric acceleration of the reaction. This may be explained on the basis of high ground state energy of the sterically crowded alcohols. Since the crowding is relieved in the product carbonyl compound as well as in the transition state leading to it, the transition state energies of the crowded and uncrowded alcohols do not differ much and steric acceleration, therefore, results.

Mechanism

The presence of a substantial primary kinetic isotope effect confirms the cleavage of α—C—H bond in the rate-determining step. The large negative value of the polar reaction constant together with substantial deuterium isotope effect indicate that the transition state approaches a carbocation in character. Hence, the transfer of hydride-ion from alcohol to the oxidant is suggested. The hydride-transfer mechanism is also supported by the major role of cation-solvating power of the solvents. The kinetics and spectral studies point to the formation of an intermediate in the pre-equilibrium. Therefore, it is likely that the oxidation involves a hydride-ion transfer via a chromate ester. This postulation is supported by an analysis of the temperature dependence of kinetic isotope effect.

Kwart and Nickle [17] have shown that a study of the dependence of the kinetic isotope effect on temperature can be gainfully employed to resolve this problem. The data for protio- and deuterio-ethanols, fitted to the familiar expression $k_H/k_D = A_H/A_D \exp(E_a/RT)$ [18,19] show a direct correspondence with the properties of a symmetrical transition state in which the activation energy difference (E_a) for k_H/k_D is equal to the zero-point energy difference for the respective C—H and C—D bonds ($\approx$ 4.5 kJ/mol) and the frequency factors and the entropies of activation



Scheme I

of the respective reactions are nearly equal. The similar phenomena were observed earlier in the oxidation of diols by BPCC [20] and that of alcohols by PFC [3].

Bordwell [21] has documented a very cogent evidence against the occurrence of concerted one-step biomolecular processes by hydrogen transfer and it is evident that in the present studies also the hydrogen transfer does not occur by an acyclic biomolecular process. It is well established that intrinsically concerted sigmatropic reactions, characterized by transfer of hydrogen in a cyclic transition state, are the only truly symmetrical processes involving a linear hydrogen transfer [22]. Littler [23] has also shown that a cyclic hydride transfer, in the oxidation of alcohols by Cr(VI), involves six electrons and, being a Huckel-type system, is an allowed process. Thus, the overall mechanism is proposed to involve the formation of a chromate ester in a fast pre-equilibrium step and then a disproportionation of the ester in a subsequent slow step via a cyclic concerted symmetrical transition state leading to the product (Scheme I). The observed hydrogen-ion dependence can be explained by assuming a rapid reversible protonation of the chromate ester (A) with the protonated ester decomposing at a rate faster than (A).

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