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OXIDATION OF SECONDARY ALCOHOLS BY ISOQUINOLINIUM DICHROMATE IN NON-AQUEOUS MEDIUM: A KINETICS STUDY

H. Jain and D. Panday✉
Department of Chemistry, Mohan Lal Sukhadiya University, Udaipur, 313001, India
✉Corresponding Author: dpanday26@gmail.com

ABSTRACT

An efficient oxidant, isoquinolinium dichromate (IQDC), was used to assess the oxidation kinetics of several secondary alcohols in a dimethylformamide medium. The reaction order for IQDC was found to be first order, whereas, for alcohols, it was less than two. The reaction was conducted at various temperatures, and the parameters for the development and disintegration of the alcohol-IQDC complexes were computed. The kinetic isotopic effect on the reaction rates was studied at various temperatures and k_H/k_D = 5.43 at 25°C suggests the primary kinetic isotope effect. [H^+] helps to catalyze the oxidation process. Multiparametric Swain's equation is used in 19 distinct solvents to assess the effect of the solvent. It has been proposed a mechanism for the oxidation process that is compatible with rate law.

Keywords: Isoquinolinium Dichromate, Kinetics, Secondary Alcohol, Oxidation.

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INTRODUCTION

The specific oxidation of alcohols to carbonyl compounds in the non-aqueous medium is an intriguing process in both theoretical and experimental areas. Alcohols may be oxidized by a number of reagents, as is well known. These oxidations and their byproducts are crucial to several biological as well as industrial production processes. A review of the literature reveals that many Cr(VI) reagents have been reported for these conversions.[1-11] The Cr(VI) [IQDC, $(C_9H_7NH^+)_2Cr_2O_7^{-2}$] complex was first described in 1998.[11] Since IQDC neither show sensitivity toward light nor deliquesce, Compared to other Cr(VI) derivatives, it may be kept more conveniently. It is widely documented that IQDC converts secondary alcohols with a yield of 60 to 90% to the corresponding carbonyl compounds.[11] No data on the oxidation of secondary alcohols by IQDC are currently available, according to a literature review. Due to the aforementioned justifications, the title reaction has been examined. The purpose of this study is to thoroughly evaluate the effects of the solvent and substituent on the oxidation process and to better comprehend the mechanism of the reaction.

EXPERIMENTAL

Material

IQDC was synthesized by reported method[6] and characterized by IR spectroscopy (KBr) which exhibited bands at 730, 765, 875, and 930 (1/cm) showing characteristic bands of $Cr_2O_7^{-2}$ ion. To confirm the purity of IQDC, iodometric titrations were employed. The secondary alcohols were received from Merck, India. The disclosed approach was used to manufacture the deuterated (^2H) propan-2-ol[12] and the purity was checked by NMR, which was 92±4%. Solvents were purified before use by method[13] reported earlier. *p*-Toluenesulphonic acid is used as [H^+] ion producer.

Product Analysis

Under kinetic conditions, *i.e.* $[propan\text{-}2\text{-}ol]_T$ kept greater to $[IQDC]_T$ (T = total concentration), 2-propanol oxidized to acetone that is confirmed by 2,4-dinitrophenylhydrazine test qualitatively and quantitatively. A solution of propan-2-ol (0.5 mol), IQDC (0.01 mol), and TsOH (0.6 mol) was prepared and added to 100 ml of DMF. This reaction mixture was left at room temperature in the dark until the reaction was completed. Distillation was used to eliminate any surplus solvent. The reaction mixture described above was saturated with a solution of 2,4-dinitrophenylhydrazine in 2M hydrochloric acid to ensure the desired result, and it was then chilled for 24 hours. The obtained product yield of DNP was 1.49g (93%) before recrystallization

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and 1.39g (88%) after. Similar tests were carried out with different secondary alcohols, and following recrystallization, the yield of the corresponding ketones ranged from 83 to 89%.

Stoichiometry

Under excess [IQDC]T over sec[propan-2-ol]T, stoichiometric studies occurred by conducting the reactions at 25°C. To establish the stoichiometry, a 100 ml solution of ($5x10^{-3}$ mol) IQDC and ($1x10^{-3}$ mol) propane 2-ol was produced in DMF with 1M TsOH. The reaction was allowed to stand for one day until it was completed. The concentration of remaining Cr(VI) was estimated by monitoring the absorbance at 370 nm. Several sets of experiments performed with distinct concentrations of alcohol and IQDC exhibited that 1 mol of IQDC was consumed by 3 mol of alcohol.

Kinetic Measurements

A UV spectrophotometer was used to track the rate at which IQDC oxidized propan-2-ol in various solvents. By maintaining $[\text{Sec. alcohol}]_T$ at least 10 times larger than $[IQDC]_T$, Under pseudo-first-order circumstances, the reactions were studied. DMF was used as the solvent unless otherwise stated. Estimating the unreacted oxidant spectrophotometrically at 370 nm at a fixed temperature (±0.1°C) for up to 70% reaction completion allowed us to monitor the progress of the reaction. Within the concentration range employed for the determination, IQDC complies with Beer's legislation. The slope of the graph ($r^2 \geq 0.999$) plotted between log [IQDC] and time was used to determine k_{obs}. The outcomes were reproduced on average three times with an error of ±3.0%.

RESULTS AND DISCUSSION

The stoichiometric estimation and product analysis showed that the oxidation of alcohol produced the equivalent ketone. The entire response might be shown by Eq.(1).

$$3R_2CHOH + (C_9H_7NH^+)_2Cr_2O_7^{-2} \longrightarrow 3R_2CO + 7H_2O + 2Cr^{+3} \quad (1)$$

Sec. alcohol IQDC Ketone

$$2\ \text{Alcohol} + \text{IQDC} \underset{}{\overset{K^{\#}}{\rightleftharpoons}} [\text{Complex}] \quad (2)$$

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

$$\text{Rate} = k_2 K^{\#} [\text{Alcohol}]^2 [IQDC]_t / (1 + K^{\#} [\text{Alcohol}]^2) \quad (4)$$

$$\text{Here, } [IQDC]_t = [IQDC] + [\text{Complex}]$$

$$\text{Rate}/[IQDC]_t = k_{obs} = \frac{k_2 K^{\#} [\text{Alcohol}]^2}{1 + K^{\#} [\text{Alcohol}]^2} \quad (5)$$

$$1/k_{obs} = \frac{1}{k_2 K^{\#}} \frac{1}{[\text{Alcohol}]^2} + \frac{1}{k_2} \quad (6)$$

Regarding IQDC, the response was determined to be first order. Moreover, k_{obs} are independent of IQDC concentration (Table-1). The order for secondary alcohols is less than two. A straight-line graph was noticed for $1/k_{obs}$ against $1/[\text{Alcohol}]^2$ (Fig.-1), this indicates that oxidation of alcohols proceeds through a complex formation between alcohol and the oxidant. This prompts the following general mechanism to be postulated by Eq.(2) - Eq.(6). The rates of oxidation of alcohols by IQDC in DMF were examined at various temperatures, and using Eq. (6), the values of decomposition constants (k_2) and formation constants ($K^{\#}$) were derived. A complex between alcohol and IQDC forms during the reaction, whose activation parameters for the breakdown and thermodynamic parameters for the creation of the complexes were evaluated at various temperatures.

Test for Free Radicals

When alcohol was being oxidized by IQDC in an inert atmosphere (which contained nitrogen), acrylonitrile was used to check for the presence of free radicals. The oxidation rate did not change when acrylonitrile was added to the reaction mixture (Table-1).

Table-1: Values of Rate Constant for IQDC Mediated Oxidation of $RCHOHCH_3$ at 25°C

10^3 [IQDC] (mol/dm^3)	[Alcohol] (mol/dm^3)	[H^+] (mol/dm^3)	R=Me	$10^4 k_{obs}$ (1/sec.)		
				R = Et	R = n-Pr	R = *i*-Bu
1.0	0.02	0.6	0.043	0.076	0.13	0.32
1.0	0.04	0.6	0.15	0.31	0.48	1.25
1.0	0.06	0.6	0.37	0.65	1.05	2.72
1.0	0.1	0.6	0.93	1.63	2.64	6.8
1.0	0.15	0.6	1.76	3.08	5.04	2.9
1.0	0.2	0.6	2.55	4.5	7.4	18.6
1.0	0.4	0.6	4.57	7.93	13.4	32.9
1.0	1.0	0.6	5.85	10.2	17.5	42.1
0.5	0.4	0.6	5.20	9.4	14.65	37.64
0.8	0.4	0.6	5.00	9.3	14.12	36.69
1.5	0.4	0.6	6.07	9.86	14.85	37.31
2.0	0.4	0.6	6.15	9.13	14.60	38.25
5.0	0.4	0.6	6.37	10.05	15.54	38.55
1.0	1.0	0.6	5.05[a]	9.28[a]	18.80[a]	40.89[a]
1.0	1.0	0.6	6.75[b]	11.85[b]	20.54[b]	41.15[b]

[a]Contained 0.01 mol/ dm^3 acrylonitrile
[b]Contained 0.05 mol/ dm^3 acrylonitrile

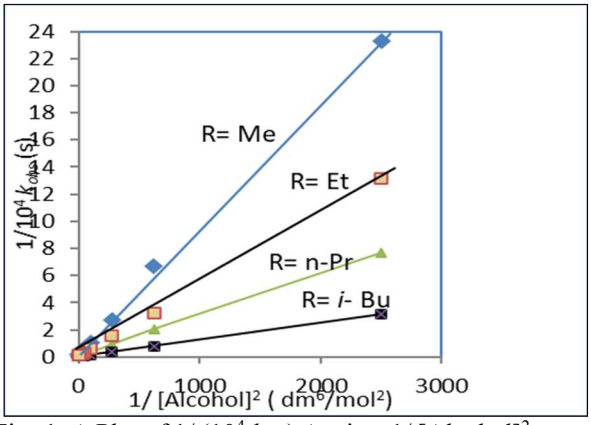


Fig.-1: A Plot of 1/ (10^4 k_{obs}) Against 1/ [Alcohol]^2 at 25°C [TsOH] = 0.6 mol/dm^3; [IQDC] = 0.001 mol/dm^3.

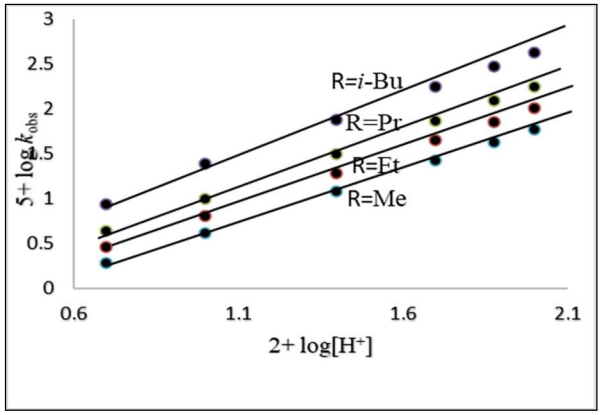


Fig.-2: A Plot of 5+ log k_{obs} Against 2+ log[H^+] at 25°C [Alcohol] = 1.0 mol/dm^3; [IQDC] = 1.0 mol/dm^3

Effect of [H^+]

The oxidation rate is found to be dependent on [H^+] concentration and rises when acidity increases, keeping the concentration of IQDC and alcohol constant. The dependence can be shown in the form of the following rate equation:

$$\text{Rate} = k[H^+]^n \qquad (\text{Where } 1<n<2)$$

A linear plot of log k_{obs} versus log [H^+] was observed with a slope between one and two, $r^2>0.99$ (Fig.-2). It was concluded that the presence of acid does not affect appreciably the formation of complex. The complex's rate of breakdown k_2, however, increases. This supports the conclusion that the complex protonates before decomposing in the slow step. Amino acid oxidation by BIDC is characterized by a similar [H^+] ion reliance.[14]

Kinetic Isotope Effect

When deuterated propan-2-ol was used in an oxidation reaction at various temperatures, it was found that the complexes of regular and deuterated alcohols have fairly comparable formation constants ($K^{\#}$), and the study of decomposition of the complex reveals that the rate of decomposition k_2 exhibits a primary kinetic isotope effect as their k_H/k_D found in a range of 4.61 to 5.43 (Table-4).

Table-2: Dependence of Secondary Alcohol [$RCHOHCH_3$] Oxidation Rate on [H^+] Concentration[a]

[H^+] (mol/dm^3)	R = Me	10^4 k_{obs} (1/sec.)R = Et	R = n-Pr	R = *i*- Bu
0.05	0.19	0.12	0.43	0.87
0.1	0.41	0.64	1.0	2.41
0.25	1.2	1.9	3.1	7.5
0.5	2.6	4.44	7.3	17.5
0.75	4.2	7.21	12.2	29.4
1.0	5.85	10.2	17.5	42.1

[a][IQDC] = 1.0 mol/dm^3

Table-3: Formation Constant Values for [$RCHOHCH_3$ – IQDC] Complex and their Thermodynamics Specification

Alcohol R	K# (dm^3/mol^1)				ΔH (kJmol^{-1})	ΔS (J$mol^{-1}K^{-1}$)	ΔG (kJmol^{-1})
	25°C	35°C	45°C	55°C			
Me	17.6	12.7	8.9	6.8	-28.6±0.5	-64±1.5	-9.6±0.36
Et	17.9	13.1	9.2	7.1	-27.5±0.6	-60±1.9	-9.6±0.45
n-Pr	16.7	11.9	8.3	6.4	-28.8±0.6	-65±1.8	-9.4±0.4
n-Bu	17.3	12.3	8.7	6.9	-27.7±50.7	-62±2.4	-9.5±0.5
i-Pr	18.2	13.4	9.6	7.5	-26.8±0.47	-58±1.5	-9.7±0.3
$ClCH_2$	17.4	12.1	8.4	6.6	-29.1±0.8	-66±2.7	-9.5±0.7
$MeOCH_2$	16.9	11.8	8.1	6.3	-29.6±0.7	-68±2.5	-9.4±0.6
i-Bu	18.1	13.3	9.4	7.2	-27.8±0.4	-61±1.46	-9.6±0.35
MeCDOHMe	17.5	12.3	8.9	7.4			

Table-4: Rate Constant Values with their Activation Specification for the Breakdown of [$RCHOHCH_3$ –IQDC] Complexes

Alcohol R	10^4 k_2 (1/sec.)				ΔH# (kJmol^{-1})	ΔS# (J$mol^{-1}K^{-1}$)	ΔG# (kJmol^{-1})
	25°C	35°C	45°C	55°C			
Me	6.2	13.6	28.4	61.9	59.5±0.8	-107±26.5	-91.4±0.65
Et	10.7	22.4	45.9	97.7	57.2±0.9	-111±3.0	-90±0.76
n-Pr	18.45	36.5	72.3	147.2	53.6±0.98	-118±3.1	-88.6±0.77
n-Bu	20.7	40.3	79.8	161	53±0.04	-119±3.4	-88.4±0.83
i-Pr	30.5	58.1	112.9	223	51±0.25	-112±3.1	-87.45±0.78
$ClCH_2$	0.01	0.028	0.067	17.0	73.6±0.7	-133±2.4	-107.2±0.05
$MeOCH_2$	0.91	2.2	4.9	11.4	65.5±0.7	-103±2.4	-96.1±0.5
i-Bu	44.3	81.9	151.5	294.2	48.6±1.1	-128±3.6	-86.5±0.8
MeCDOHMe	1.14	2.63	5.83	13.4	63.9±0.87	106±2.8	-95.5±0.68
k_H/k_D	5.43	5.17	4.87	4.61			

Table-5: Solvent Impact on IQDC's Oxidation of propan-2-ol at 25°C.

Solvent	K# (dm^3/mol)	10^4 k_2 (1/sec)	Solvent	K# (dm^3/mol)	10^4 k_2 (1/sec)
Chloroform	18.1	3.27	Cyclohexane	18.2	0.12
Carbondisulphide	16.8	0.46	Toluene	17.6	0.94
1,2- Dichloroethane	17.5	3.95	Acetophenone	18.0	4.95
Dichloroethane	18.2	3.8	Tetrahydrofurane	16.9	1.72
DMSO	17.7	12.5	Acetone	16.9	5.5
Tert-Butyl alcohol	16.3	1.31	Dioxane	18.5	1.78
Dimethylformamide	17.6	6.2	1,2-Dimethoxyethane	17.8	0.90
Butanone	16.6	2.5	Ethanoic acid	18.6	0.59
Nitrobenzene	18.4	4.6	Ethyl ethanoate	17.2	1.31
Benzene	16.5	1.2			

Exner's graph[15] between log k_2 (at 25°C) and log k_2 (at 55°C) was found straight line (r^2 = 0.9995) for the eight alcohols. The isokinetic temperature for the oxidation reactions is 689 ± 8K. At this temperature, the

oxidant IQDC oxidizes all the substrates with a similar mechanism. From the perspective of proton transfer, oxidant IQDC seems to be an ionic molecule. To assume that IQDC is still undissociated in our studies, the conductivity data were adequate. Following the addition of IQDC to DMF, very little change in conductivity was noticed. Further, the rate of oxidation was studied after the addition of isoquinolinium ion and reached to postulate that IQDC works as unionized. Protonation of IQDC takes place and produces an electrophile which increases the rate of oxidation. Hydrogen-ion dependency can be summarized in the following chemical equation.

$$\underset{(A)}{(IQOH)_2Cr_2O_5} + 2\,H^+ \overset{K_1}{\rightleftharpoons} \underset{(B)}{(IQOH)_2O[OCr^+(OH)]_2} \qquad (7)$$

Here, IQ stands for isoquinolene and the further impact of hydrogen ion concentration on oxidation rate is best summarized by

$$1/k_{obs} = a + b/[H^+]^2 \qquad (8)$$

Solvent Effect

The findings of a study on the influence of solvents on the oxidizing process of propan-2-ol by IQDC are summarized in Table-5, which shows the formation constant, $K^{\#}$, is essentially the same in all nineteen solvents. Still, the decomposition constant, k_2, varied significantly. The effect of solvent polarity was studied on the rate constant of oxidation, the rate constant k_2 in eighteen different solvents was correlated by using the Kamlet and Taft model.[16] Here Carbon disulfide was not included as a solvent because complete information of solvent parameter is not available. The outcomes of the application of the Kamlet multiparametric equation indicated the influence of the solvent on the oxidation process is in poor correlation.

Swain's method[17] was also applied to illustrate the solvent effect.

$$\log k = aA + bB + C \qquad (9)$$

Where A and B represent the solvating power of anion and cation respectively, and A+B is defined to reflect the solvent polarity with C as an intercept. Here in Swain's equation 19 solvents effects were studied.

$$\log k_2 = (0.64 \pm 0.06)\,A + (1.81 \pm 0.04)\,B - 5.07 \qquad (10)$$

$R^2 = 0.9909$, SD = 0.04, $\psi = 0.1008$

$$\log k_2 = (0.39 \pm 0.60)\,A - 3.83 \qquad (11)$$

$r^2 = 0.0243$, SD = 0.48, $\psi = 1.0148$

$$\log k_2 = (1.76 \pm 0.12)\,B - 4.8 \qquad (12)$$

$r^2 = 0.9246$, SD = 0.13, $\psi = 0.2821$

$$\log k_2 = (1.42 \pm 0.15)\,(A+B) - 5.03 \qquad (13)$$

$r^2 = 0.8363$, SD = 0.19, $\psi = 0.4156$

The findings demonstrated a significant connection with Swain's equation (9). The solvent impact was influenced by both the solvating capacities of cations and anions. However, the solvent's cation-solvating capacity plays a significant role and accounts for about 92% of the data. A+B, which represents the solvent polarity, was responsible for almost 84% of the data. The relative permittivity of the solvents was correlated with the data obtained from the solvent effect. However, there was a nonlinear relationship between log k and the inverse of relative permittivity, ($r^2 = 0.4900$). This demonstrated that the relative permittivity and polarity of the solvent do not reflect the same solvent properties.

Correlation Analysis of Reactivity

A study of the complex creation constants($K^{\#}$) of the alcohol-IQDC complexes for different substituted alcohols allows us to conclude that $K^{\#}$ is insensitive to changing substituent (Table-2) however, significant

changes in the rate of breakdown (k_2) of complexes were observed. A satisfying correlation was not observed with either Taft's polar substituent constant[18] (σ*) or steric substituent constant (E_s) separately for rate constant k_2.

$$\log k_2 = (-2.16 \pm 0.28)\ \sigma^* - 2.89 \quad (14)$$
$r^2 = 0.9050$, SD = 0.3467, n = 8 , $\psi = 0.3295$, T = 25°C

$$\log k_2 = (-1.4 \pm 0.69)\ E_s - 3.66 \quad (15)$$
$r^2 = 0.4234$, SD = 0.8546, n = 8, $\psi = 0.8177$, T = 25°C

The dual substituent equation of Pavelich –Taft[18] was used to correlate oxidation rates of the alcohols.

$$\log k = \rho^*\sigma^* + \delta\ E_s + \log k_0 \quad (16)$$

Table-6: The Influence of Temperature on Reaction Constants

T/ °C	ρ^*	δ	R^2	SD	ψ	N
25°C	-1.87±0.001	-0.71±0.003	0.9999	0.001	0.011	8
35°C	-1.76±0.004	-0.63±0.002	0.9983	0.004	0.047	8
45°C	-1.68±0.003	0.58±0.001	0.9911	0.097	0.10	8
55°C	-1.61±0.002	-0.53±0.005	0.9942	0.005	0.087	8

N = number of data points

The observed data were compiled in Table-6, which revealed a strong negative connection between reaction constants. Alcohols that are sterically crowded have high ground state energy, which manifests as negative steric reaction parameters and steric acceleration of reaction rates. In order to avoid a major change in the sterically more and less crowded alcohol's transition phase energies, Crowding has been released between the product's ketone and the associated transition state. As a result, steric acceleration occurs. The negative polar reaction constant serves as a marker for the carbocation formation in the slow step's transition state.

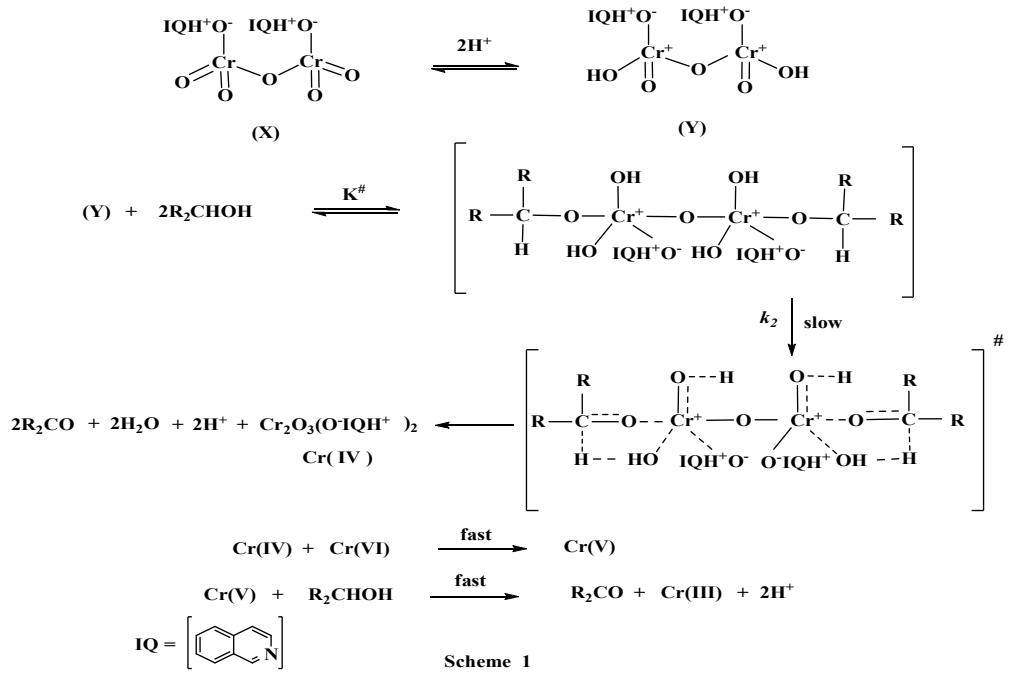
Mechanism

Following tests using acrylonitrile, there is no chance of a single electron transferring here. In the quick equilibrium stage, the stoichiometry of the current reaction is 2:1 with regard to alcohol and IQDC. The constants ($K^{\#}$) of the formation of initially formed complexes of regular and deuterated secondary alcohols were found to be highly comparable, and research on the complex's breakdown shows that the rate of breakdown displays a strong primary kinetic isotope effect. This led to the conclusion that the rate-controlling phase is where the cleavage of the α-C-H bond occurs. The breaking of this α-C-H bond as a hydride-ion during the rate-regulating phase results in the emergence of electron-poor species in the stage of transition. It is further supported by the solvent effect's observed correlation study. The mechanism is proposed with the support of all the stoichiometry data. The intended product is produced when the intermediate complex accepts two protons from the acid by an H^- ion transfer from the substrate to IQDC. which then undergoes breakdown (Scheme-1).

For the transfer of hydride ions, either an acyclic or cyclic process may be envisaged. Kwart and Nickle[20], who discovered that the isotopic effect on kinetics depends on temperature, may also be taken into account. The data for ordinary and deuterated propane 2-ol can be shown by the following equation:

$$k_H/k_D = (A_H/A_D)\ e^{(-\Delta Ea/RT)} \quad (17)$$

Here ΔE_a is the variation in activation energy of k_H and k_D is nearly comparable to 4.5 kJ/mol with $\Delta S^{\#}$ being almost the same for both.[21,22] The hydrogen shift in the reaction takes place *via* a cyclic transition phase and the evidence in favor of a concerted mechanism of one-step bimolecular hydrogen shifting is also given by Bordwell.[23] Sigmatropic reactions are followed by cyclic transition state demand hydrogen transfer linearly in a concerted manner symmetrically.[24] Littler[25] has also laid out that cyclic H^--ion deportation, alcohols being oxidized by Cr(VI), includes 6 e^-s, which is an allowed process according to the Huckel $(4n+2)\pi\ e^-$ system, is a recognized method. Thus, the H^--ion transfer occurs through a cyclic transition phase by producing a cyclic chromate ester in this oxidation. Initially hydrogen atom of the -OH group is transferred to IQDC, leading to the emergence of a chromate ester followed by protonation.



Scheme 1

In the transition state before the Unimolecular decomposition of ester, hydrogen is simultaneously attached to both carbon of alcohol as well as -OH group. The route of the e-flow in a concerted cyclic phase of transition passes via the carbon-hydrogen-oxygen-chromium link as the H atom is transported as an H^- ion. This would permit the origin of the final product by conducting an electron across the Cr-O-C connection. Different substituents each have a unique impact on the rate of the reaction, which is what causes a negative reaction constant. The two opposing charged ends of the solvent molecule become heavily solvated as a result of the charge segregation in the oxidant transition phase, which makes the solvent molecule less mobile and reduces entropy. It is crucial to note that while Cr(VI) reduces to Cr(IV) before reducing to Cr(III), Cr(VI) does not decrease Cr(III) directly. It is anticipated that it will mix with another species of Cr(VI) to form Cr(V), which will be swiftly transformed to become Cr(III). Such a process of oxidation has been thoroughly documented.[26]

CONCLUSION

The reaction of secondary alcohol was found in first order with respect to IQDC. Formation of a carbocation in the transition state of the rate-determining step takes place. A hydride shift in reaction takes place via a cyclic transition state.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

D. Panday http://orchid.org/0000-0002-8096-4672
H. Jain http://orchid.org/0009-0006-4419-9842

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