

Oxidation Of Inositol By Cerium (IV) In Acidic Medium:- A Kinetic Study”.

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Abstract: The oxidation of inositol by cerium(IV) has been studied experimentally in the acidic medium at fixed ionic strength and temperature. The reaction showed a first order dependence with respect to [Ce(IV)] and less than 1st order kinetics in both [H⁺] and [INOS]. By increasing the values of both ionic strength and dielectric constant of the reaction medium which increases the rate of oxidation reaction. The addition of Ce(III) products to the reaction mixture does not show any significance or increase in the rate of reaction. The oxidation product of inositol has been identified by the chemical reactions and tools and spectroscopically inosose. The compatible mechanism for the oxidation reaction has been put forward and the rate law associated with the reaction mechanism has been derived. The activation energy has been determined experimentally.

Key words: Inositol, Inosose, Kinetic mechanism, Oxidation, Rate constant, Cerium(IV)

Date of Submission: 28-01-2021

Date of Acceptance: 12-02-2021

I. Introduction:

Inositol is an important B-complex. It is six membered polyol which is synthesized in the kidneys. It is widely distributed in plants and animal kingdom. It is also present in the muscles and organs of the animals. It is growth promoting factor which possess in antibaldness property. From the previous researches it is found that it is a powerful insecticides also. Inositol and some of its derivatives are used as the basis for some signaling and secondary messenger molecule which are involved in insulin signal transduction give expression, breakdown of fats and reducing the blood cholesterol.

The kinetic mechanism of oxidation of inositol was investigated by different oxidants like alkaline potassium periodate, alkaline diperiodato- cuprate(III) and alkaline permanganate. Vanadium(V) in acidic medium and by chromic acid and sulphuric acid.

Cerium(IV) is a significant one electron oxidant in acidic medium. In perchlorate medium this oxidant has not used yet or rarely used. It is due to presence of dimeric and polymers of cerium (IV) in perchlorate medium. The oxidation of cerium (IV) in perchloric acid solution takes place much faster than those in sulphuric acid. It is evident from the number of investigations on kinetic of oxidation of inositol by various oxidants has been performed. There is a lack of literatures on kinetics of poly of compound by cerium(IV) in acidic medium. This observation prompted us to investigate the above mentioned reaction in the title. The main objective of the present study to check the reaction rate of inositol towards cerium (IV) in acidic medium (perchloric acids).

II. Experimental Methodology:

Materials used:

All the chemicals used in this experiment were of laboratory reagent grade and the solutions were prepared by dissolving the required amount of the samples in distilled water. The stock of inositol solution was prepared by dissolving the proper amount of the recrystallized samples in distilled water. The fresh solution of cerium (IV) has been prepared by dissolving the ceric ammonium sulphate in sulphuric acid diluted with distilled water and kept for overnight. The solution of cerium (IV) has been kept in a dark glass bottle for 24 hours. Cerium(III) solution has been also prepared by dissolving cerium (III) acetate in distilled water.

KINETIC MECHANISM:

The kinetic measurement were followed under Pseudo First order reaction condition with an excess of inositol over cerium (IV) has been used during the course of reaction it was monitored by the decrease in absorption of cerium(IV) with respect to time $\lambda=318\text{nm}$ at its maximum absorbance whereas the other constituent of the reaction mixture does not have significant absorption at this wave length as mentioned in the figures given below.

The absorbance has been measured by the UV-VIS-NIR3600 double beam spectrophotometer. The

Pseudo first order rate constant (k_{obs}) were calculated with graph as $\ln(\text{absorbance})$ versus time. The rate constants were the mean of at least three kinetic measurements. The rate constants were reproducible to 3-4%. The spectral changes for the oxidation of inositol by cerium(IV) in perchlorate medium as shown in figure. The scanned spectra indicate gradual decay of cerium(IV) at its maximum absorption with respect to time resultantly the reduction of cerium(IV) by inositol reductant takes place. The reaction order with respect to the reactants were determined from the slopes of $\log k(\text{obs.})$ versus $\log(\text{concentrated perchlorate})$ plots by varying the concentration of inositol substrate and perchloric acid while keeping other constant.

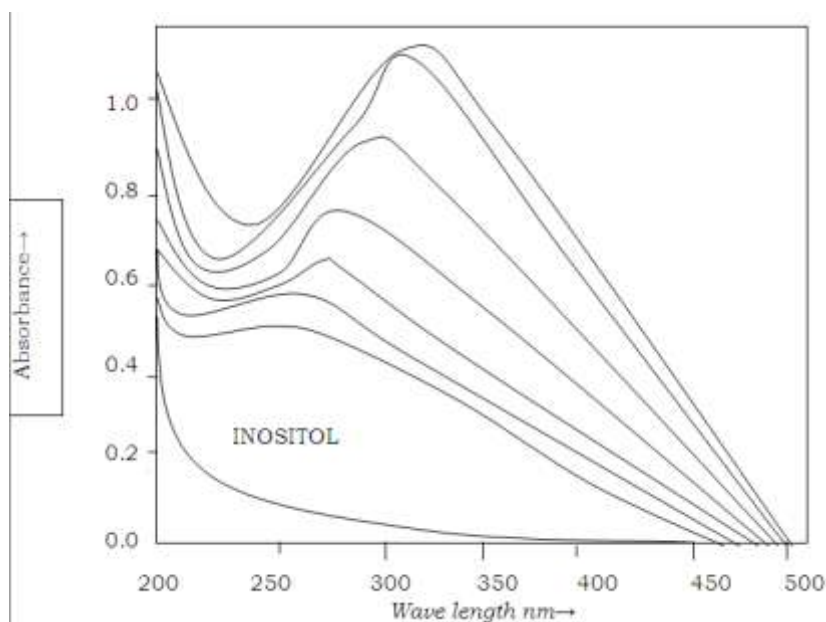
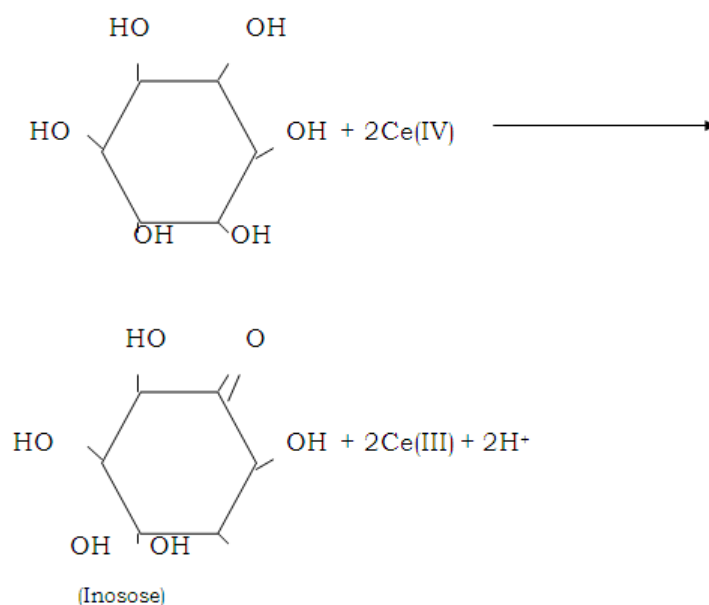


Figure :- Spectral changes during oxidation of inositol by Ce(IV) in perchlorate medium $[\text{INOS}] = 5.0 \times 10^{-3}$
 $[\text{CeIV}] = 2.0 \times 10^{-4}$, $[\text{H}^+] = 0.5$ avl $= 1.0 \text{ mol dm}^{-3}$ at 25°C (RT) Time interval = 3min.

Results:

Stoichiometry and product analysis:

Reaction mixture containing different quantities of Cerium(IV) and inositol at constant $[\text{H}^+]$. Ionic strength and temperature are allowed to take part in reaction for 24 hours. For the oxidation the remaining unconsumed Ce(IV) was determined by the spectrometer at 314nm. The results indicated that two moles of Ce(IV) are unconsumed by one mole of inositol and the final oxidation products.



The above stoichiometry shows the consistency with the results of product analysis which is carried out by both spectroscopically and chemically. The oxidation product of inositol was identified as the monoketone derivative as inosose which has been confirmed by the addition of 2,4-dinitrophenyl hydrative to the reaction mixture

Rate dependence of [Ce (IV)]:

The obs first order rate constant K(obs) at determined at different concentrations of Ce(IV) keeping other variables constant. It has been observed that increasing concentration of Ce(IV) oxidants does not change the oxidation rate of inositol. This shows that oxidation rate is independent of oxidants concentration. Concentration and the order of reaction with respect to the oxidant is first order.

Rate Dependence of Inositol:

The reductant inositol varied in the range of 2.0×10^{-3} mol dm⁻³ to 1.0×10^{-3} mol dm⁻³. while other variables are kept constant. It has been observed that the increasing inositol concentration increases the oxidation rates. From the graph plotted between Kobs versus [inositol] was found to be linear positive intercept which confirms the less than unit with respect to [inositol].

Rate dependance on [H+]:

The effect of Hydrogen ion concentration on the oxidation rate has been studied by varying the perchloric acid concentration in the range of (0.1-0.9 mol dm⁻³) and keeping the concentration of all other reactants constant.

Effect of ionic strength and dielectric constants:

The effect of ionic strength of the reaction medium on the oxidation rate has been studied by varying ionic strength in the range of 1.0-3.0 mol dm⁻³ using potassium perchlorate as an electrolyte keeping the concentration of all other reactants constant. The results confirms that increasing ionic strength of the medium increases the oxidation rate and the Debye-Huckel plot was linear with the positive slope of 0.94.

And the effects of dielectric constants (D) has also been studied by the varying contents of acetic acid and water. The reaction mixture with all other conditions being kept constant. The rate constant clearly decreases as D of the solvent being decreased.

Table: Effect of variation of [Ce(IV)][INOS][H⁺] and ionic strength I on the first order rate constant value k_{obs} in the oxidation rate of inositol by Ce(IV)in perchlorate medium at room temperature.

10 ⁴ Ce(IV)	mol dm ⁻³	10 ³ [INOSITOL]	mol dm ⁻³	H ⁺ mol dm ⁻³	I mol dm ⁻³
0.5		6.0	0.5	1.0	42.4
1.0		6.0	0.5	1.0	41.93
2.0		6.0	0.5	1.0	42.82
3.0		6.0	0.5	1.0	43.14
4.0		6.0	0.5	1.0	19.25
2.0		2.0	0.5	1.0	33.0
2.0		4.0	0.5	1.0	42.84
2.0		6.0	0.5	1.0	56.55
2.0		8.0	0.5	1.0	71.38
2.0		10.0	0.5	1.0	13.24
2.0		6.0	0.3	1.0	32.62
2.0		6.0	0.5	1.0	42.76
2.0		6.0	0.7	1.0	67.43
2.0		6.0	0.9	1.0	86.01
2.0		6.0	0.5	1.0	42.79
2.0		6.0	0.5	1.5	44.71
2.0		6.0	0.5	2.0	47.02
2.0		6.0	0.5	2.5	50.01
2.0		6.0	0.5	3.0	52.42

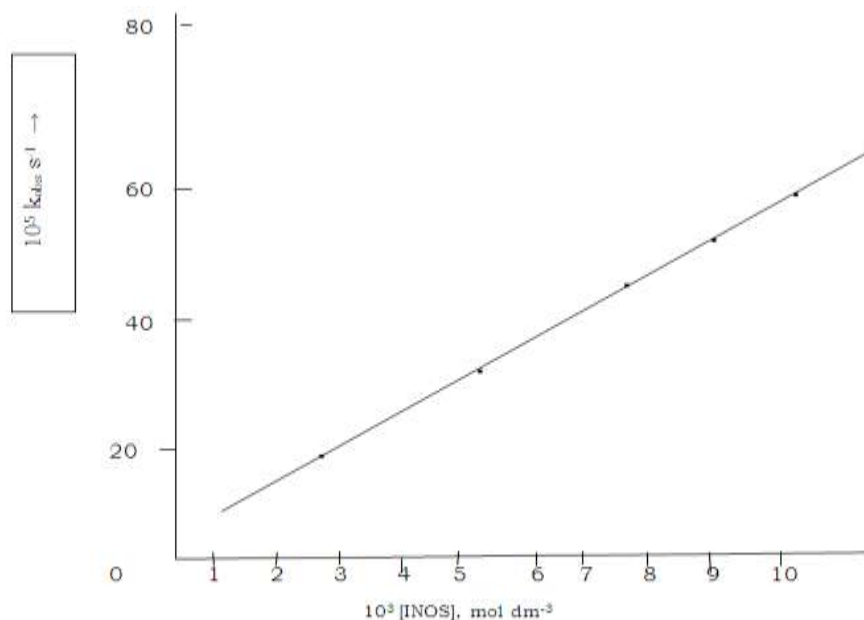


Figure:2 Plot between k_{obs} vs $[\text{INOS}]$
 $[\text{Ce(IV)}] = 2.0 \times 10^{-4}$, $[\text{H}^+] 0.5$ & $I = 1.0 \text{ mol dm}^{-3}$ at 25°C

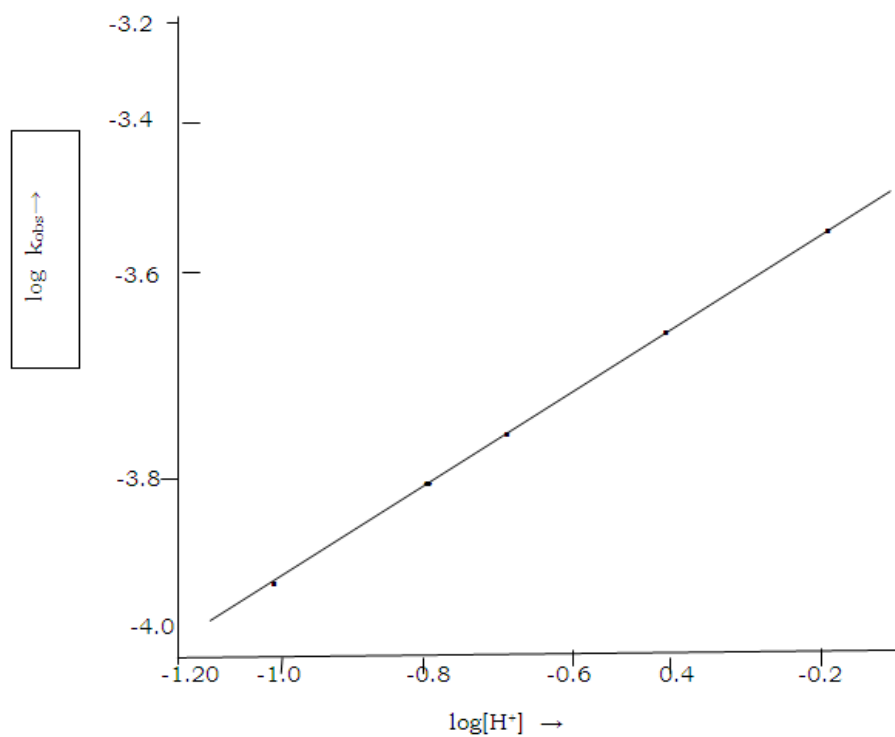


Figure 3: plot of $\log k_{\text{obs}}$ versus $\log[\text{H}^+]$ in the oxidation
of inositol by Ce(IV) in perchlorate medium
 $[\text{INOS}] = 6.0 \times 10^{-4}$ and $I = 1.0 \text{ mol dm}^{-3}$ at 25°C

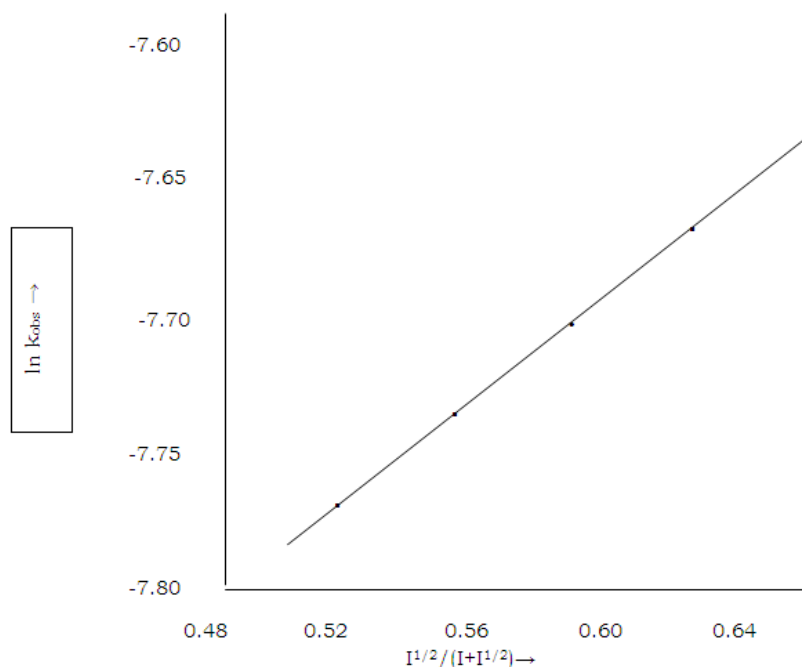


Figure: 4 Effect of ionic strength on k_{obs} in the oxidation of inositol by Ce(IV) in perchlorate medium [INOS] = 5.0×10^{-3} and Ce(IV) = 2.0×10^{-4} mol dm^{-3} at 25°C

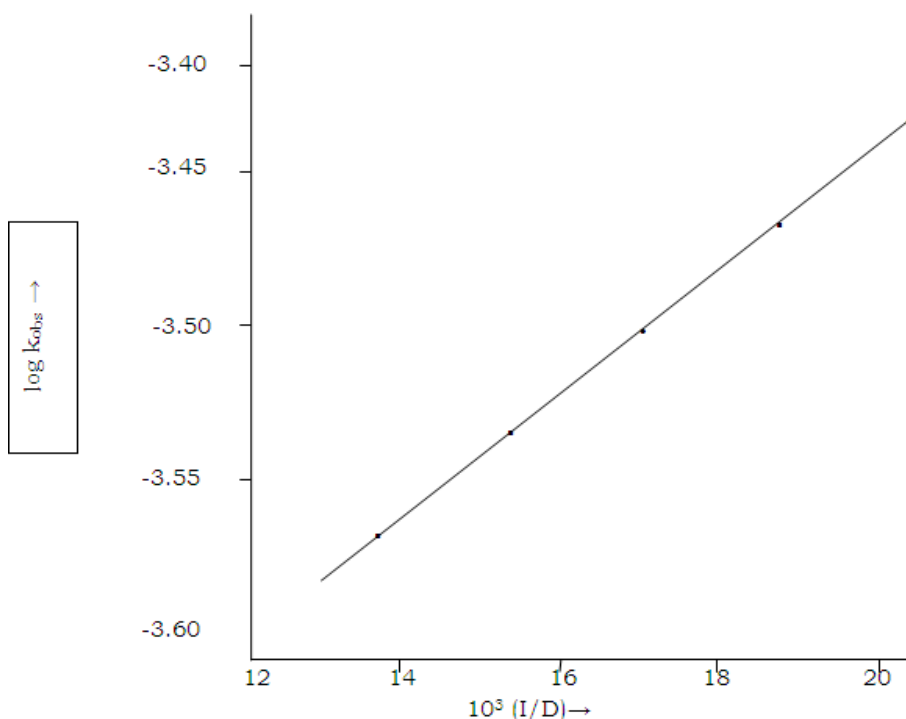


Figure:5 Effect of dielectric constant of the reaction medium on k_{obs} in the oxidation of inositol by Ce(IV) in perchlorate medium [INOS] = 6.0×10^{-3} and Ce(IV) = 2.0×10^{-4} mol dm^{-3} at 25°C

Effects of initial products taken

The effects of added cerium (III) product has been studied in the concentration range 1.0-6.0x at constant concentrations of the oxidants reductants and acid . It was found that Ce (IV) has no significant on oxidation rate of Inositol .

Effects of Temperature

The oxidation rate has been observed at five different temperatures in the range of 288 to 308 K at constant concentrations of the reactants and other conditions being constant. The activation parameters of the second order on rate constant (K_2) and calculate using Arrhenius and Eyring plots and show in the table given below
Activation parametres of second order rate constant (K_2) in the oxidation of Inositol by Ce(IV) in perchlorate medium .

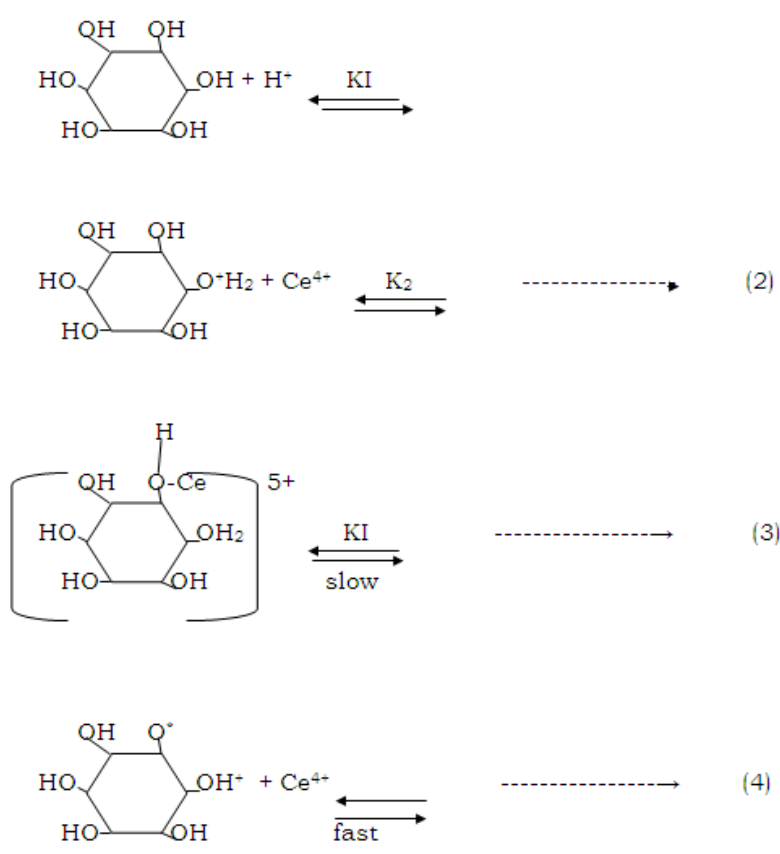
[INOS]= 6.0×10^{-3} ,Ce (IV)= 2.0×10^{-4}
[H^+]=0.5 and I= 1.0 mol dm^{-3}

Polymerisation

In the reaction mixture a known quantity of acrylonitrile has been added and kept in inert atmosphere for 10 hours. On diluting the reaction mixture with methanol white precipitate is obtained, seems to be generation of free radical in the reaction when the experiment has been repeated in the absence of Inositol under similar conditions. The test was negative. It shows that the reaction took place under free radical mechanism.

III. Discussion:

It has been observed that active species of Ce(IV) in acidic medium were found to be free monomeric species of Ce^{4+} . Its hydrolysed forms are $Ce(OH)^{3+}$, $Ce(OH)_2^{2+}$ or partially dimeric species $(Ce-O-Ce)^{6+}$ and $(HO-Ce-O-CeOH)^{4+}$. However, spectrophotometric study indicates the hydrated forms of dimers are the more predominant species at $[H^+] > 1.0 \text{ mol dm}^{-3}$ up to the concentration of $1.5 \times 10^{-3} \text{ mol dm}^{-3}$ of the Ce(IV) whereas the hydrolyzed forms and dimers are more predominant at $[H^+] < 0.8 \text{ mol dm}^{-3}$. So under the experimental conditions increasing the oxidation rate with the increase in $[H^+]$ regards Ce^{4+} is the more active form of Ce(IV) than the hydrolysed form. The reaction between Inositol and Ce(IV) in a perchlorate medium have stoichiometry of 1:2 Inos :Ce(IV). The reaction is of first order dependence with respect to [Ce IV] less than unit order dependence with respect to $[H^+]$ and [INOS]. Owing to the experimental results a possible reaction mechanism has been proposed for which all the observed orders in each constituents may be well accommodated. The proposed mechanism involves protonation of inositol in the first step followed by the complex formation of protonated Inositol with kinetically active species of Cerium (IV) oxidant. Ce^{4+} prior to rate determining step. Such complex decomposes to form free radical intermediate. Species further reacts with another species of oxidants in a fast step to form the final oxidation step of Inositol (INOSOSE) as shown below:





The suggested mechanism leads to the following rate law expression:

$$\text{Rate} = \frac{k_1 K_1 K_2 [\text{Ce(IV)}] [\text{INOS}] [\text{H}^+]}{1 + k_1 [\text{H}^+] + K_1 K_2 [\text{INOS}] [\text{H}^+]} \quad (6.)$$

The rate law (6.) is consistent with all the observed order with respect to different species.

Under Pseudo 1st order the rate law can be expressed by equation (7):

$$\text{Rate} = d[\text{Ce(IV)}] / dt \quad (7.)$$

Comparing equation (6) & (7)

$$K_{\text{obs.}} = \frac{k_1 K_1 K_2 [\text{Ce(IV)}] [\text{INOS}] [\text{H}^+]}{1 + k_1 [\text{H}^+] + K_1 K_2 [\text{INOS}] [\text{H}^+]} \quad (8.)$$

By rearranging the equation (8):

$$1/K_{\text{obs.}} = \left[\frac{1 + K_1 [\text{H}^+]}{k_1 K_1 K_2 [\text{H}^+]} \right] \times 1/[\text{INOS}] + 1/K_1 \quad (9.)$$

$$1/K_{\text{obs.}} = \left[\frac{1}{k_1 K_1 K_2 [\text{INOS}]} \right] \times 1/[\text{H}^+] + 1/K_1 K_2 [\text{INOS}] + 1/k_1 \quad (10.)$$

Equation (9)&(10) requires the plot of 1/K_{obs} versus 1/[INOS] at constant [H⁺] and 1/K_{obs} versus [H⁺] at constant [INOS] to be taken with +ve intercept.

IV. Conclusions:

The oxidation of inositol by cerium(IV) in acidic medium has been studied experimentally & spectrophotometrically. The oxidation product of Inositol was identified as Inosose. The mechanistic scheme for the oxidation reaction has been proposed. The activation parameters of the second order rate constant were calculated and discussed.

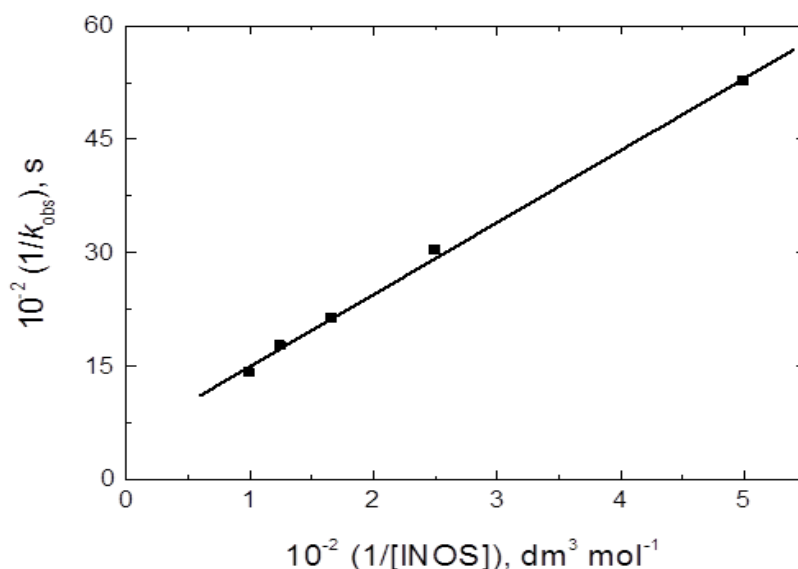


Figure 6. Plot of 1/k_{obs} against 1/[INOS] in the oxidation of inositol by Ce(IV) in perchlorate medium. [Ce(IV)] = 2.0 x 10⁻⁴, [H⁺] = 0.5 and I = 1.0 mol dm⁻³ at 25 °C.

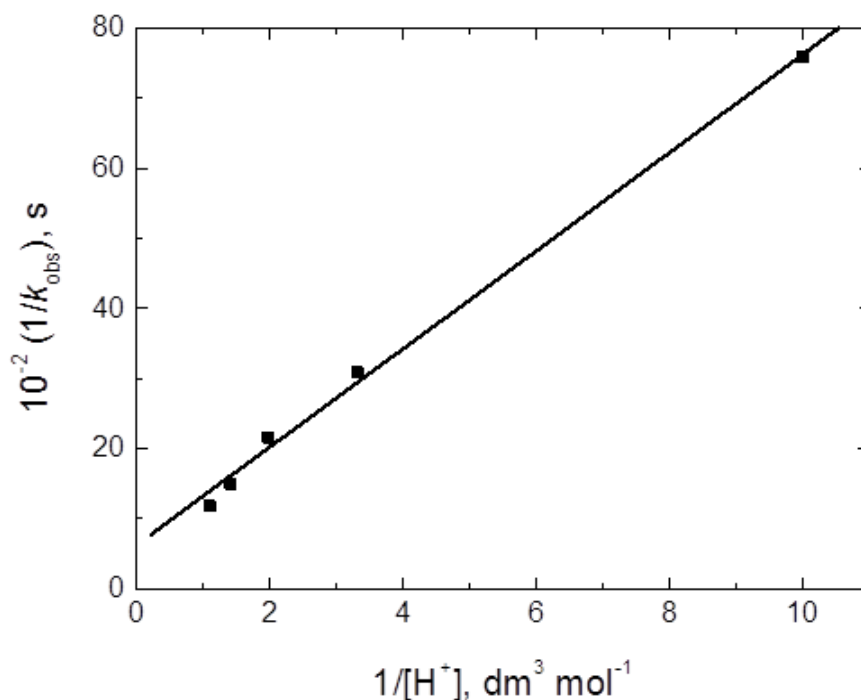


Figure 7. Plot of $1/k_{\text{obs}}$ against $1/[H^+]$ in the oxidation of inositol by Ce(IV) in perchlorate medium. $[INOS] = 6.0 \times 10^{-3}$, $[Ce(IV)] = 2.0 \times 10^{-4}$ and $I = 1.0 \text{ mol dm}^{-3}$ at 25°C .

Derivation of the rate-law expression:

According to the suggested mechanistic Scheme 1,

$$\text{Rate} = \frac{-d[Ce(IV)]}{dt} = k_1[C] \text{-----} \rightarrow (A1)$$

$$K_1 = \frac{[INOS^+]}{[INOS][H^+]},$$

$$[INOS^+] = K_1[INOS][H^+] \text{-----} \rightarrow (A2)$$

$$K_2 = \frac{[C]}{[INOS^+][Ce^{4+}]},$$

$$[C] = K_2[INOS^+][Ce^{4+}] \text{-----} \rightarrow (A3)$$

Substituting Eq. (A2) into Eq. (A3) yields,

$$[C] = K_1K_2[INOS][Ce^{4+}][H^+] \text{-----} \rightarrow (A4)$$

Also, substituting Eq. (A4) into Eq. (A1) gives,

$$\text{Rate} = k_1K_1K_2 [INOS] [Ce^{4+}] [H^+] \text{-----} \rightarrow (A5)$$

The total concentration of inositol is given by,

$$[INOS]_T = [INOS]_F + [INOS^+] + [C] \text{-----} \rightarrow (A6)$$

Where ‘T’ and ‘F’ refer to total and free concentrations.

Substituting Eq. (A2) and (A4) into Eq. (A6) gives,

$$[INOS]_T = [INOS]_F + K_1 [INOS] [H^+] + K_1K_2 [INOS] [Ce^{4+}] [H^+] \text{-----} \rightarrow (A7)$$

$$[INOS]_T = [INOS]_F(1 + K_1[H^+] + K_1K_2[Ce^{4+}][H^+]) \text{-----} \rightarrow (A8)$$

$$[INOS]_F = \frac{[INOS]_T}{1 + K_1[H^+] + K_1K_2[Ce^{4+}][H^+]} \text{-----} \rightarrow (A9)$$

Therefore,

Also, the total concentration of cerium (IV) equals to,

$$[\text{Ce(IV)}]_{\text{T}} = [\text{Ce}^{4+}]_{\text{F}} + [\text{C}] \quad \text{-----} \rightarrow (\text{A10})$$

$$[\text{Ce(IV)}]_{\text{T}} = [\text{Ce}^{4+}]_{\text{F}} + K_1 K_2 [\text{INOS}] [\text{Ce}^{4+}] [\text{H}^+] \quad \text{-----} \rightarrow (\text{A11})$$

$$[\text{Ce(IV)}]_{\text{T}} = [\text{Ce}^{4+}]_{\text{F}} (1 + K_1 K_2 [\text{INOS}] [\text{H}^+]) \quad \text{-----} \rightarrow (\text{A12})$$

$$[\text{Ce}^{4+}]_{\text{F}} = \frac{[\text{Ce(IV)}]_{\text{T}}}{1 + K_1 K_2 [\text{INOS}] [\text{H}^+]} \quad \text{-----} \rightarrow (\text{A13})$$

Due to high $[\text{H}^+]$, $[\text{H}^+]_{\text{T}} = [\text{H}^+] \quad \text{-----} \rightarrow (\text{A14})$

Substituting Eqs. (A9), (A13) and (A14) into Eq. (A5) (and omitting 'T' and 'F' subscripts) gives,

$$\text{Rate} = \frac{k_3 K_1 K_2 [\text{Ce(IV)}] [\text{INOS}] [\text{H}^+]}{(1 + K_1 K_2 [\text{INOS}] [\text{H}^+]) (1 + K_1 [\text{H}^+] + K_1 K_2 [\text{Ce}^{4+}] [\text{H}^+])} \quad \text{-----} \rightarrow (15)$$

Owing to low $[\text{Ce}^{4+}]$, the last denominator term in Eq. (A15) is neglected.

Therefore,

$$\text{Rate} = \frac{k_3 K_1 K_2 [\text{Ce(IV)}] [\text{INOS}] [\text{H}^+]}{(1 + K_1 K_2 [\text{INOS}] [\text{H}^+]) (1 + K_1 [\text{H}^+])} \quad \text{-----} \rightarrow (\text{A16})$$

$$\text{Rate} = \frac{k_3 K_1 K_2 [\text{Ce(IV)}] [\text{INOS}] [\text{H}^+]}{1 + K_1 [\text{H}^+] + K_1 K_2 [\text{INOS}] [\text{H}^+] + K_1^2 K_2 [\text{INOS}] [\text{H}^+]^2} \quad \text{-----} \rightarrow (\text{A17})$$

Due to low $[\text{INOS}]$, the denominator term, $K_1^2 K_2 [\text{INOS}] [\text{H}^+]^2$, in Eq. (A17) can be neglected, therefore,

$$\text{Rate} = \frac{k_3 K_1 K_2 [\text{Ce(IV)}] [\text{INOS}] [\text{H}^+]}{1 + K_1 [\text{H}^+] + K_1 K_2 [\text{INOS}] [\text{H}^+]} \quad \text{-----} \rightarrow (\text{A18})$$

Under pseudo-first order condition, the rate-law can be expressed by equation (A15),

$$\text{Rate} = \frac{-d[\text{Ce(IV)}]}{dt} = k_{\text{obs}} [\text{Ce(IV)}] \quad \text{-----} \rightarrow (\text{A19})$$

Comparing Eqs. (A18) and (A19),

$$\text{Rate} = \frac{k_3 K_1 K_2 [\text{INOS}] [\text{H}^+]}{1 + K_1 [\text{H}^+] + K_1 K_2 [\text{INOS}] [\text{H}^+]} \quad \text{-----} \rightarrow (\text{A20})$$

and with rearrangement, the following equations are obtained,

$$\frac{1}{k_{\text{obs}}} = \left(\frac{1 + K_1 [\text{H}^+]}{k_3 K_1 K_2 [\text{H}^+]} \right) \frac{1}{[\text{INOS}]} + \frac{1}{k_3} \quad \text{-----} \rightarrow (\text{A21})$$

$$\frac{1}{k_{\text{obs}}} = \left(\frac{1}{k_3 K_1 K_2 [\text{INOS}]} \right) \frac{1}{[\text{H}^+]} + \frac{1}{k_3 K_2 [\text{INOS}]} + \frac{1}{k_3} \quad \text{-----} \rightarrow (\text{A22})$$

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Sanjeev Kumar Sinha, et. al. "Oxidation Of Inositol By Cerium (IV) In Acidic Medium:- A Kinetic Study".
IOSR Journal of Applied Chemistry (IOSR-JAC), 14(2), (2021): pp 07-16.