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Kinetics and Mechanism of Electron Transfer to Platinum(IV) by Cadaverine in Perchloric Acid Medium

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Abstract

The kinetics of platinum(IV) oxidation of cadaverine (CAD) in perchloric acid medium was studied at a constant ionic strength of 2.0 mol dm^{-3} and at 25°C. The oxidation reaction was followed spectrophotometrically. The reaction exhibited a first order kinetics in [Pt^{IV}] and less than unit order dependences with respect to [CAD] and [H^{+}]. Increasing ionic strength and dielectric constant decreased the oxidation rate. The final oxidation products of cadaverine were identified as 5-aminopentanal and ammonia. The oxidation mechanism was proposed and the appropriate rate-law expression was deduced. The activation parameters of the second order rate constant were evaluated and discussed.

1. Introduction

Polyamines are widely distributed in living organisms and are known to be essential elements for normal growth and development. Cadaverine (1,5-pentanediamine) is a foul-smelling toxic diamine compound produced by protein hydrolysis during putrefaction of animal tissue [1]. It is produced in small quantities by living beings. It is partially responsible for the distinctive odors of urine. The odor commonly associated with bacterial vaginosis has been linked to cadaverine and putrescine [2].

Platinum(IV) complexes has remarkable anticancer properties [3-6]. They appeared attractive because they are usually substitution inert and require reduction to Pt^{II} species to act as potential anticancer drugs. The anticancer activity of platinum(IV) complexes may be due to effective Pt^{IV} transport into the cell followed by reduction to the more reactive platinum(II) compounds. Several kinetic investigations on the oxidation of inorganic and organic substrates using platinum(IV) complexes in the form of hexachloroplatinate(IV) as an oxidant, were performed in the last decades [7-24]. Hexachloroplatinate(IV) may behave as one or two electron oxidant, depending upon the substrate and experimental conditions. The literature survey reveals that there were no reports on the kinetics of oxidation of cadaverine by any oxidant. Hence, we have investigated the title reaction in order to understand the complicated biological reactions in living systems and to propose a plausible oxidation mechanism.

2. Materials and Methods

2.1. Materials

All chemicals employed in this work were of reagent grade and their solutions were prepared by dissolving the requisite amounts of the samples in doubly distilled water. A stock solution of cadaverine was prepared by dissolving the sample (Merck) in doubly distilled water. Chloroplatinic acid solution (Johnson Matthey) was used without further purification. Required solution of the oxidant was freshly prepared before each experiment by proper dilution of its original solution which was standardized spectrophotometrically [25].

2.2. Kinetic Measurements

The kinetic runs were carried out under pseudo-first order conditions with cadaverine concentration being greater than the platinum(IV) concentration. The ionic strength of the reaction medium was maintained constant by addition of sodium perchlorate as an inert electrolyte. The progress of the reaction was followed by measuring the decrease in absorbance of platinum(IV), as a function of time, at its absorption maximum, 263 nm. The absorption measurements were made in a thermostatted Shimadzu UV-VIS-NIR-3600 double-beam spectrophotometer. The applicability of Beer's law was verified at 263 nm under the reaction conditions that molar extinction coefficient was determined ($\varepsilon = 13133 \pm 261$ $dm^3\ mol^{-1}\ cm^{-1}$) and was found to be in a good agreement with that reported previously [25].

The kinetic runs were followed for more than 80% completion of the reaction. The observed-first order rate constant (k_{obs}) was determined from the gradients of ln(absorbance) versus time plots, by considering the initial straight line region. The rate constants were reproducible to within ±3% and were the average of atleast two independent kinetic runs. The reaction orders with respect to the reactants were determined from the slopes of log k_{obs} versus log(Conc.) plots by varying the concentrations of cadaverine and perchloric acid, in turn, while keeping other conditions constant. The spectral changes during oxidation of cadaverine by platinum (IV) in perchloric acid medium are shown in Fig. 1. The figure shows a gradual decrease in platinum (IV) absorbance at its absorption maximum with time as a result of its reduction to platinum (II) by cadaverine.

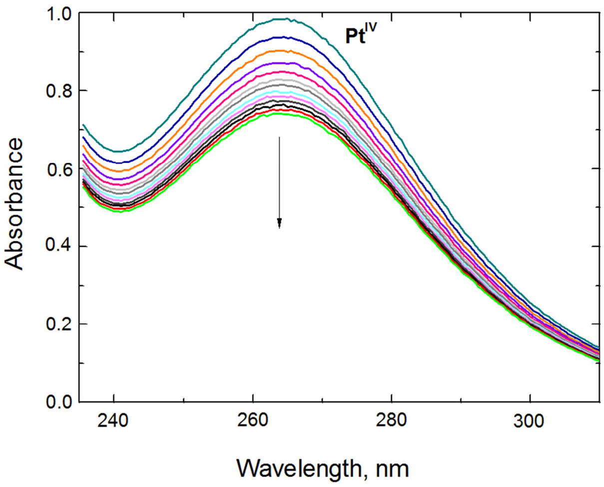

Figure 1. *Spectral changes in the oxidation of cadaverine by platinum(IV) in perchloric acid medium. [CAD] = 6.0 x10^{-3}, [Pt^{IV}] = 8.0 x10^{-5}, [H$^+$] = 1.0 and I = 2.0 mol dm^{-3} at 25 °C. Scan time interval = 2.0 min.*

3. Result and Discussion

3.1. Stoichiometry and Products Analysis

Various ratios of the reaction mixtures containing cadaverine and platinum (IV) were mixed at constant acidity and ionic strength, then equilibrated for 24 h in an inert atmosphere. Estimation of the unconsumed [Pt^{IV}] was carried out spectrophotometrically. The results showed that one mole of platinum(IV) consumed one mole of cadaverine according to the following stoichiometric equation,

$$H_2N(CH_2)_5NH_2 + [PtCl_6]^{2-} + H_2O$$
$$\rightarrow H_2N(CH_2)_4CHO + NH_3 + [PtCl_4]^{2-} + 2Cl^- + 2H^+ \quad (1)$$

where $H_2N(CH_2)_5NH_2$ and $H_2N(CH_2)_4CHO$ are cadaverine and its corresponding aldehyde (5-aminopentanal), respectively. The product aldehyde was identified by liquid chromatography (HPLC) and by spot test [26], and was also estimated quantitatively as its 2,4-dinitrophenylhydrazone derivative [27], and ammonia was identified by Nessler's reagent [27].

3.2. Reaction Rate Dependence on [Pt^{IV}]

To examine the effect of platinum (IV) oxidant on the reaction rate, the concentration of platinum (IV) was varied in the range of (4.0 – 14.0) x 10^{-5} mol dm^{-3} at constant [CAD], [H$^+$], ionic strength and temperature. The non-variation in the observed-first order rate constant at various concentrations of platinum (IV) (Table 1) indicates that the order with respect to the oxidant is confirmed to be one.

3.3. Reaction Rate Dependence on [CAD]

The observed-first order rate constant was determined at different initial concentrations of the reductant cadaverine while keeping all other conditions constant. The results showed that the observed rate constant increased with increasing concentration of cadaverine as listed in Table 1. A plot of the observed rate constant versus [CAD] was linear with a positive intercept confirming less than unit order dependence with respect to cadaverine (Fig. 2).

3.4. Reaction Rate Dependence on [H$^+$]

The oxidation rate was measured at constant [CAD], [Pt^{IV}], ionic strength and temperature but with various [H$^+$] (0.4 – 1.9 mol dm^{-3}). The oxidation rate was found to increase as [H$^+$] increased with less than unit order dependence as the slope of log k_{obs} versus log [H^+] plot (slope = 0.71), Fig. 3.

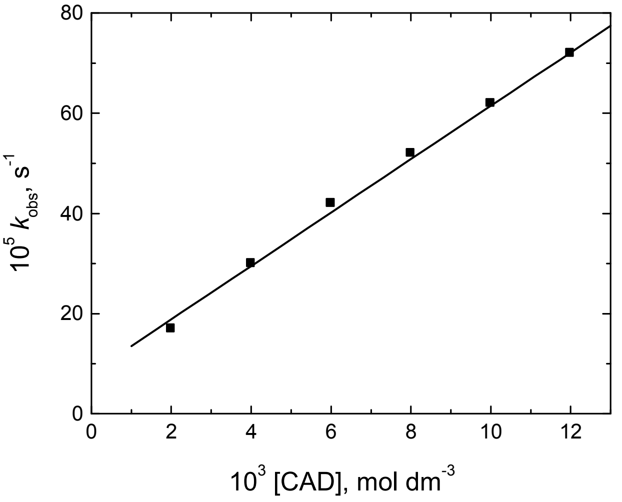

Figure 2. *Plot of the observed-first order rate constant (k_{obs}) versus [CAD] in the oxidation of cadaverine by platinum(IV) in perchloric acid medium. [Pt^{IV}] = 8.0 x 10^{-5}, [H^+] = 1.0 and I = 2.0 mol dm^{-3} at 25°C.*

Table 1. *Effect of variation of [Pt^{IV}], [CAD], [H^+] and ionic strength, I, on the observed-first order rate constant (k_{obs}) in the oxidation of cadaverine by platinum(IV) in perchloric acid medium at 25°C.*

10^5 [Pt^{IV}] (mol dm^{-3})	10^3 [CAD] (mol dm^{-3})	[H^+] (mol dm^{-3})	*I* (mol dm^{-3})	$10^5 k_{\mathrm{obs}}$ (s^{-1})
4.0	6.0	1.0	2.0	41.6
6.0	6.0	1.0	2.0	40.9
8.0	6.0	1.0	2.0	42.1
10.0	6.0	1.0	2.0	43.6
12.0	6.0	1.0	2.0	42.3
14.0	6.0	1.0	2.0	42.8
8.0	**2.0**	1.0	2.0	16.9
8.0	**4.0**	1.0	2.0	30.2
8.0	**6.0**	1.0	2.0	42.1
8.0	**8.0**	1.0	2.0	52.0
8.0	**10.0**	1.0	2.0	62.3
8.0	**12.0**	1.0	2.0	71.8
8.0	6.0	**0.4**	2.0	22.0
8.0	6.0	**0.7**	2.0	33.2
8.0	6.0	**1.0**	2.0	42.1
8.0	6.0	**1.3**	2.0	49.9
8.0	6.0	**1.6**	2.0	59.0
8.0	6.0	**1.9**	2.0	67.6
8.0	6.0	1.0	**2.0**	42.1
8.0	6.0	1.0	**2.5**	39.2
8.0	6.0	1.0	**3.0**	37.1
8.0	6.0	1.0	**3.5**	34.3
8.0	6.0	1.0	**4.0**	30.7
8.0	6.0	1.0	**4.5**	28.5

Experimental Error ± 3%

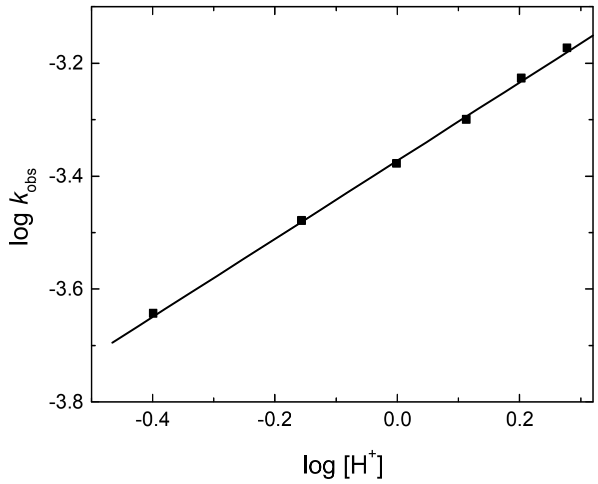

Figure 3. *Plot of log k_{obs} versus log [H^+] in the oxidation of cadaverine by platinum(IV) in perchloric acid medium.[CAD]= 6.0 x 10^{-3}, [Pt^{IV}] = 8.0 x 10^{-5} and I = 2.0 mol dm^{-3} at 25°C.*

3.5. Effect of Ionic Strength and Dielectric Constant

The effect of ionic strength on the oxidation rate was investigated by varying the concentration of sodium perchlorate in the reaction medium at constant concentrations of cadaverine and platinum(IV), and at constant pH and temperature. It was found that the observed-first order rate constants decreased with increase in the ionic strength of the medium, and the Debye-Hückel plot was linear with a negative slope (Fig. 4). Furthermore, the effect dielectric constant (*D*) was studied by varying the acetic acid – water content (0 - 40%) in the reaction mixture with all other conditions being constant. The results revealed that the oxidation rate decreased with increasing dielectric constant of the reaction medium and the plot of log k_{obs} versus 1/*D* was linear with a positive slope as shown in Figure 5.

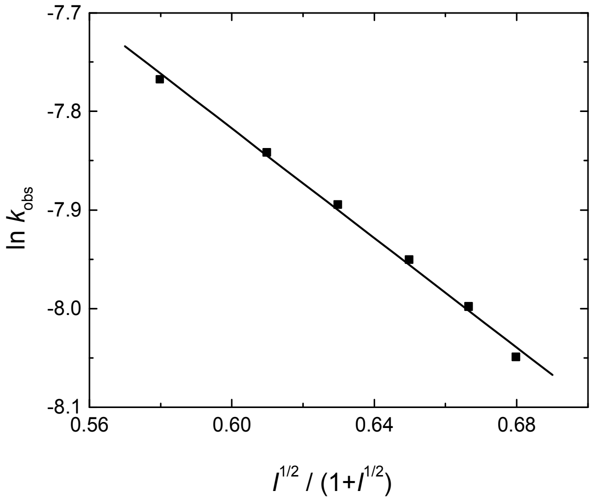

Figure 4. *Debye–Hückel plot in the oxidation of cadaverine by platinum(IV) in perchloric acid medium. [CAD]= 6.0 x 10^{-3},[Pt^{IV}] = 8.0 x 10^{-5}, [H^+] = 1.0 and I = 2.0 mol dm^{-3} at 25°C.*

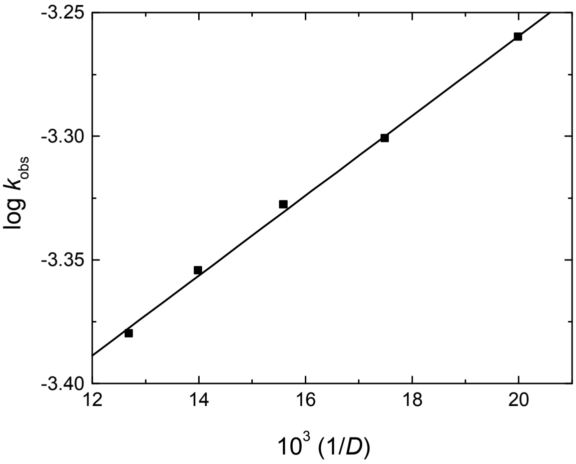

Figure 5. *Plot of log k_{obs} versus 1/D for the oxidation of cadaverine by platinum(IV) in perchloric acid medium.[CAD]= 6.0 x 10^{-3}, [Pt^{IV}] = 8.0 x 10^{-5}, [H^+] = 1.0 and I = 2.0 mol dm^{-3} at 25°C.*

3.6. Effect of Temperature

In order to determine the activation parameters, the oxidation reaction was investigated at five different temperatures in the range of 15 – 35 °C. The oxidation rate was found to increase with raising temperature. The activation parameters of the second order rate constants (k_2) were calculated using Arrhenius and Eyring plots and were listed in Table 2.

Table 2. *Activation parameters of the second order rate constants k_2 in the oxidation of cadaverine by platinum(IV) in perchloric acid medium. [CAD] = 6.0 x 10^{-3}, [Pt^{IV}] = 8.0 x 10^{-5}, [H^+] = 1.0 and I = 2.0 mol dm^{-3}.*

$\Delta S^{\neq}$, J mol^{-1}K^{-1}	$\Delta H^{\neq}$, kJ mol^{-1}	$\Delta G^{\neq}_{293}$, kJ mol^{-1}	$E_a^{\neq}$, kJ mol^{-1}
-97.08	37.43	66.36	39.78

3.7. Polymerization Test

The intervention of free radicals during the oxidation reaction was investigated by polymerization test. The reaction mixture to which a known quantity of acrylonitrile scavenger was initially added and was kept for 24 h in an inert atmosphere. On diluting the mixtures with methanol, no white precipitate was formed thus confirming the absence of free radical intervention in the reaction.

3.8. Reaction Mechanism

It was reported [28] that platinum(IV) species exist as $[PtCl_6]^{2-}$ in acid medium, which assumed to be the principal reactive oxidant. Reduction of $[PtCl_6]^{2-}$ generally proceeds according to the equation: $[PtCl_6]^{2-} + 2e^- = [PtCl_4]^{2-} + 2Cl^-$.

In this redox process, octahedral Pt^{IV} is reduced to square planner Pt^{II} with release of two Cl^- ions. Therefore, this reaction is better classified as reductive-elimination reaction [29]. There are two suggested alternative reaction mechanisms for the oxidation by hexachloroplatinate (IV). The first mechanism involves a simultaneous two-electron transfer in a single step. The second one involves two successive one-electron transfer steps. If the transition states of reductant and/or oxidant are unstable, a simultaneous two-electron transfer mechanism may be suggested such as that in the case of oxidation of uranium (IV) by $[PtCl_6]^{2-}$ [30]. In the present study, addition of acrylonitrile monomer to the reaction mixture failed to give polymerized products. It may be that the free radical like Pt^{III} species is too short-lived to interact with acrylonitrile to give the polymerized product under our experimental conditions. Consequently, the two-electron transfer mechanism seems plausible.

The present reaction between cadaverine and platinum(IV) in perchloric acid medium has a stoichiometry of 1:1. The reaction exhibited a first order kinetics on [Pt^{IV}], less than unit order dependences with respect to [CAD] and [H^+]. The observed enhancement of the oxidation rate upon increasing acid concentration with less than unit order dependence suggests that the protonated form of cadaverine substrate (CAD^+) may be considered as the kinetically reactive species in the rate-determining step, which play the main role in the reaction kinetics. The less than unit order dependences with respect to cadaverine concentrations suggests formation of an intermediate complex between cadaverine and platinum(IV) prior to the slow step. Complex formation was proved kinetically [31] by the non-zero intercept of the plot of $1/k_{obs}$ versus 1/[CAD] (Fig. 6). Also, decreasing the oxidation rate with increasing ionic strength and dielectric constant of the reaction medium confirms that the reaction in the rate-determining step occurred between two ions with unlike charge [32, 33], i.e. between $[PtCl_6]^{2-}$ and CAD^+. The failure of the spectrophotometric detection of such intermediate complex (Fig. 1) may be interpreted by either lower concentration of the reactants used and, hence, the expected lower absorbitivity of the formed complex and/or the fast subsequent decomposition of the intermediate in comparison to its formation.

Owing to the experimental results and the above-mentioned arguments, the suggested oxidation mechanism involves protonation of cadaverine in the first step followed by combination of protonated cadaverine (CAD^+) with the kinetically active species of platinum(IV), $[PtCl_6]^{2-}$, to form an intermediate complex (C). Such complex slowly decomposes to give rise to the oxidation product of cadaverine as depicted by the following equations,

$$H_2N(CH_2)_5NH_2 + H^+ \underset{}{\overset{K_1}{\rightleftharpoons}} H_3N^+(CH_2)_5NH_2 \quad (CAD^+) \tag{2}$$

$$H_3N^+(CH_2)_5NH_2 + [PtCl_6]^{2-} \overset{K_2}{\rightleftharpoons} [H_3N(CH_2)_5NH_2.\ PtCl_6]^- \quad (C) \tag{3}$$

$$[H_3N(CH_2)_5NH_2.\ PtCl_6]^- + H_2O \xrightarrow[slow]{k_1} H_2N(CH_2)_4CHO + NH_3 + [PtCl_4]^{2-} + 2Cl^- + 2H^+ \tag{4}$$

The suggested mechanism leads to the following rate-law expression,

$$\text{Rate} = \frac{-d[\text{PtCl}_6{}^{2-}]}{dt} = \frac{k_1K_1K_2[\text{PtCl}_6{}^{2-}][\text{CAD}][\text{H}^+]}{(1+K_1[\text{H}^+]+K_1K_2[\text{PtCl}_6{}^{2-}][\text{H}^+])(1+K_1K_2[\text{CAD}][\text{H}^+])} \quad (5)$$

In view of low concentration of $[PtCl_6]^{2-}$ used, the term $K_1K_2[PtCl_6^{2-}][H^+]$ in the denominator of Eq. (5) can be neglected. Therefore, Eq. (5) becomes

$$\text{Rate} = \frac{k_1K_1K_2[\text{PtCl}_6{}^{2-}][\text{CAD}][\text{H}^+]}{(1+K_1[\text{H}^+])(1+K_1K_2[\text{CAD}][\text{H}^+])} \quad (6)$$

The rate law is consistent with all the observed orders with respect to different species.

Under pseudo-first order condition

$$\text{Rate} = \frac{-d[\text{PtCl}_6{}^{2-}]}{dt} = k_{\text{obs}}[\text{PtCl}_6{}^{2-}] \quad (7)$$

Comparing Eqs. (6) and (7) and rearrangement, we obtain,

$$\frac{1}{k_{\text{obs}}} = \left(\frac{1+K_1[\text{H}^+]}{k_1K_1K_2[\text{H}^+]}\right)\frac{1}{[\text{CAD}]} + C \quad (8)$$

where $C = (1+K_1[H^+])/k_1$.

According to Eq. (8), the plot of $1/k_{obs}$ versus 1/[CAD], at constant $[H^+]$, and $1/k_{obs}$ versus $1/[H^+]$, at constant [CAD], should be linear with positive intercepts on the $1/k_{obs}$ axes as were observed experimentally as shown in Figures 6 and 7, respectively.

On the other hand, the obtained negative value of $\Delta S^{\neq}$ indicates that there is a decrease in the randomness during the oxidation process. This leads to the formation of compacted intermediate complex and such activated complex is more ordered than the reactants due to loss of degree of freedom [34]. Again, the negative values of both $\Delta H^{\neq}$ and $\Delta G^{\neq}$ indicate the exothermic formation of the intermediate and its spontaneity, respectively.

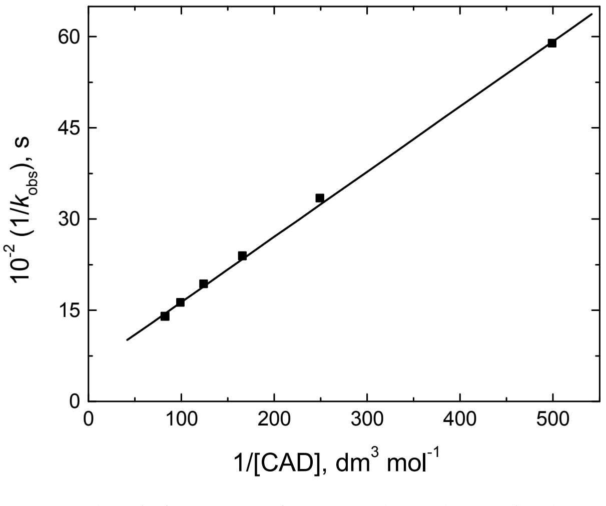

Figure 6. *Plot of $1/k_{obs}$ versus 1/[CAD] in the oxidation of cadaverine by platinum(IV) in perchloric acid medium. $[Pt^{IV}] = 8.0 \times 10^{-5}$, $[H^+] = 1.0$ and $I = 2.0$ mol dm^{-3} at 25°C.*

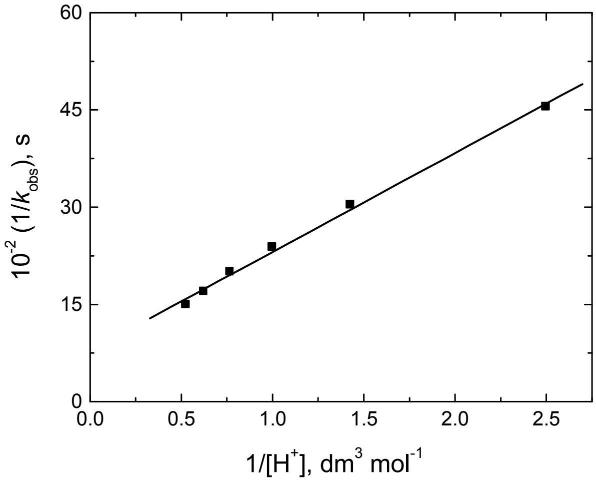

Figure 7. *Plot of $1/k_{obs}$ versus $1/[H^+]$ in the oxidation of cadaverine by platinum(IV) in perchloric acid medium. $[CAD] = 6.0 \times 10^{-3}$, $[Pt^{IV}] = 8.0 \times 10^{-5}$ and $I = 2.0$ mol dm^{-3} at 25°C.*

4. Conclusion

The kinetics of oxidation of cadaverine by platinum(IV) in perchloric acid medium was studied spectrophotometrically. The final oxidation products of cadaverine were identified as 5-aminopentanal and ammonia. The oxidation mechanism was proposed and the appropriate rate-law expression was deduced. The activation parameters of the second order rate constant were evaluated and discussed.

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