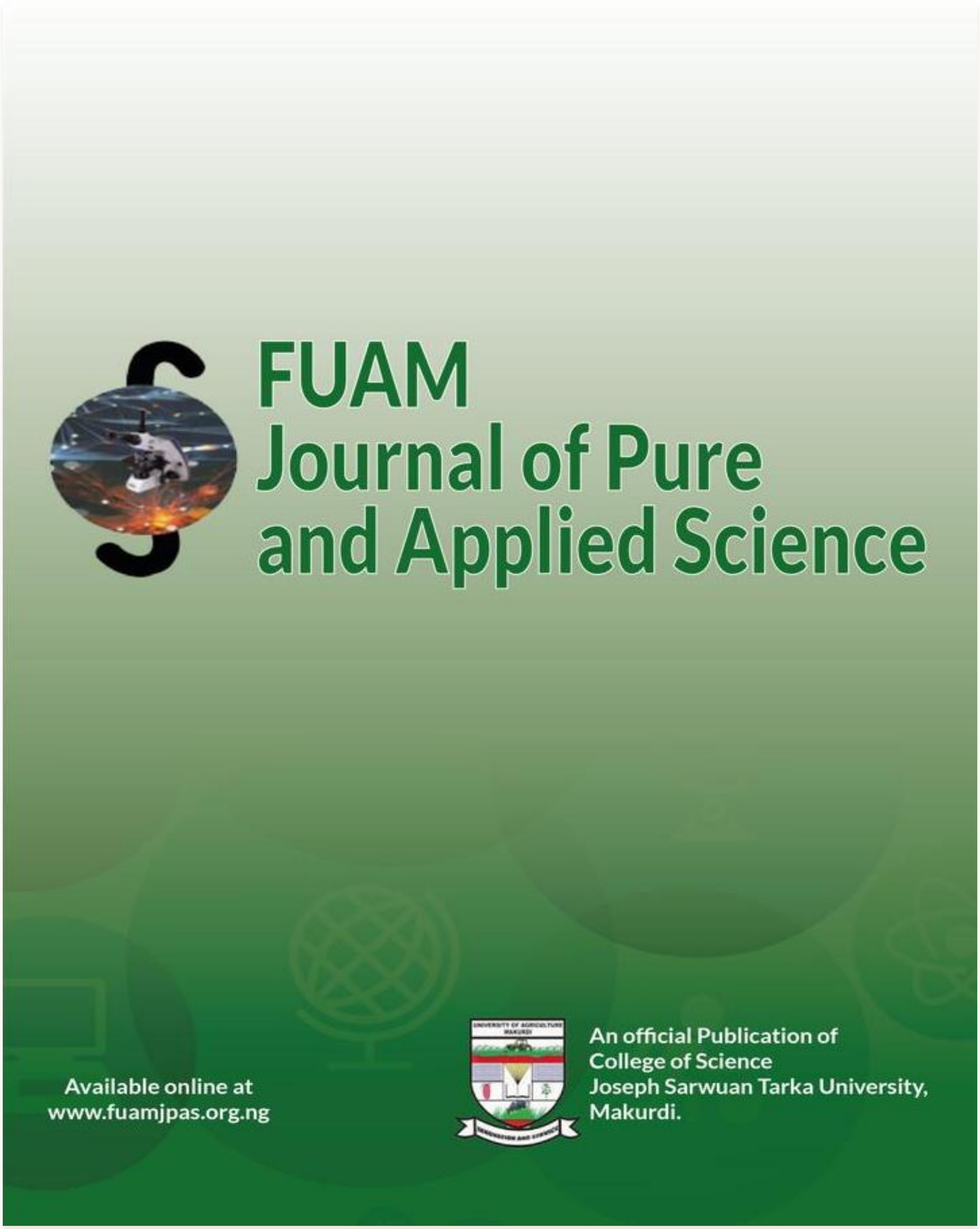

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Kinetics, Thermodynamics and Mechanistic Investigation of the Reaction between $AuCl_3(OH)]^-$ and Propanol in Acidic Medium

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Abstract
Reaction of Gold(III) complex ion, $[AuCl_3(OH)]^-$, with propanol (PrOH) was studied spectrophotometrically in perchloric acid medium at ionic strength (μ) = 0.05 mol dm^{-3} ($NaClO_4$) and T = 30.0 ± 1 °C. Stoichiometry of the reaction revealed that one mole of $[AuCl_3(OH)]^-$ was reduced by one mole of propanol. The rate of reaction was first order in $[AuCl_3(OH)^-]$ as well as in $[C_3H_7OH]$ with a second order overall, having mean second order rate constant of 0.8649 ± 0.061 dm^3 mol^{-1} s^{-1}. Addition of acid within the range 5.0×10^{-4} to 1.0×10^{-2} mol dm^{-3} decreased the rate of reaction from 0.940 to 0.307 dm^3 mol^{-1} s^{-1}. Increasing μ from 2.0×10^{-2} to 1.2×10^{-1} mol dm^{-3} ($NaClO_4$) had no effect on the rate of the reaction. The same trend was observed on varying dielectric constant from 78.40 to 73.59. Michaelis – Menten's type plot was linear and with negligible intercept. Entropy of activation was found to be -206.56 $JK^{-1}mol^{-1}$ while activated enthalpy was 3.668 kJ mol^{-1}. Au(I) was the product of $[AuCl_3(OH)]^-$ reduction while propanal was obtained from propanol oxidation. FTIR spectral showed band for propanal formation (C=O) at 1764 cm^{-1}. Based on negligible intercept from Michaelis – Menten's type plot and the absence of spectroscopically determinable intermediate complex, the reaction was proposed to have occurred by outer-sphere mechanistic route.

Keywords: Gold(III) complex ion, Propanol, Kinetics, Thermodynamics, Mechanism

Introduction

Gold is a metallic material with a distinct yellow colour and metallic lustre. It has an atomic number of 79 and atomic mass of 196.967. Au(0) has the electronic configuration $[Xe]\,4f^{14}5d^{10}6s^1$, it is regarded as a transition metal at higher oxidation states and a member of the copper triad, copper, silver and gold. It is the most noble of all metals and this is the key to both its eternal romance and its many practical uses in industry. Gold has an electrochemical potential which is the lowest of any metal. This means that gold in any cationic form will accept electrons from virtually any reducing agent (Equation 1) with standard reduction potential of 1.40 V explains that at under standard conditions, the cationic gold(III) is reduced to the +1oxidation state [1].

$$Au^{3+} + 2e^- \longrightarrow Au^+ \qquad (1)$$

The redox reaction of gold(III) has been found to be the major driving force of its application in diverse fields. The development of gold(III) complexes has been encouraged by the fact that an increase in the number of nitrogen donor atoms in the coordination sphere of gold(III) leads to a significant decrease in the reduction potential. It is important to note that for almost all known active gold(III) complexes, the active metabolites could be gold(I) species produced by Gold(III) reduction *in vivo*. Reduction potential values are an unavoidable parameter in establishing the mode of action since most metallopharmaceuticals are activated by *in vivo* electron transfer [2].

Electron transfer reaction of metal ions and their complexes is a key chemical process in nature [3]. These reactions are useful in industries for the utilization of these complexes as homogeneous catalysts and in chelate therapy in medicinal and environmental chemistry [4]. Chelate therapy in cancer treatment had relied on platinum-based anti-cancer agents, this has been documented to be plagued with some toxicities. Current research interest includes the development of new metal based anti-tumor agents having pharmacological activities that are different from platinum-based anti-tumor agents [5]. Among this class, gold(III) complexes are considered to possess pharmacodynamic and pharmacokinetic features different from platinum-based drugs, but have powerful inhibitory properties on cell growth [6]. The anti-tumor properties of the gold(III) metal center were combined with the potential chemo-protective function of coordinated dithiocarbamates in order to reduce toxic side effects such as nephrotoxicity induced by clinically established platinum-based drugs [7].

Since gold(III) complexes hold much promise as anti-tumor agents, several of them have been synthesized and applied in cancer therapy and targeted drug delivery. Furthermore, gold(I) drugs may be activated *in vivo* to gold(III) metabolites under physiological conditions. However, the intrinsic mechanisms driving these physiological processes have not been fully unraveled. This paucity of kinetic and mechanistic data has limited applications of gold(III) derived drugs. This paper presents the kinetic, thermodynamic and mechanistic approaches of the reduction of gold(III) complex ion with propanol. It is envisaged that our results will unravel the mechanisms driving the speciation of gold compounds under physiological conditions.

Materials and Methods

Analytical grade chemicals were used as supplied without further purification. $HAuCl_4.3H_2O$ [Sigma–Aldrich, 99 %] was the oxidizing agent, C_3H_7OH [BDH, 99.8 %] was the reducing agent, $HClO_4$ [Sigma –Aldrich, 70 %] was used to alter acidity level of reaction, $NaClO_4$ [Sigma–Aldrich, 98 %] was used to adjust reaction ionic strength. C_3H_5NO was used to test for the presence of free radical in the reaction. CH_3COONa and $KClO_4$ were used to investigate catalysis or otherwise of the reaction. All solutions of reagents were prepared in deionized water.

Stoichiometry

Stoichiometry of the reaction was determined spectrophotometrically by titrations employing mole ratio method [8] under the conditions of $[H^+] = 1.0 \times 10^{-3}$ mol dm^{-3}, $\mu = 0.02$ mol dm^{-3} ($NaClO_4$), $[AuCl_3(OH)]^- = 1 \times 10^{-4}$ mol dm^{-3}, $[C_3H_7OH] = (2.5 \times 10^{-5}$ to $2.5 \times 10^{-4})$ mol dm^{-3}, Temperature (T) = 30.0 ± 1.0 °C and λmax = 310 nm. The point of inflexion from plot of absorbance versus mole ratio of the reactants gave the stoichiometry of the reaction.

Kinetic measurements

All kinetic measurements were performed under pseudo-first-order conditions, where $[C_3H_7OH]$ were >> $[AuCl_3(OH)]^-$ at T = 30.0 ± 1.0 °C, $[H^+] = 1.0 \times 10^{-3}$ mol dm^{-3} and $\mu = 0.05$ mol dm^{-3}. The course of reaction was monitored by measuring change in absorbance of $[AuCl_3(OH)]^-$ at 310 nm as a function of time on a JENWAY 6405 Uv-vis spectrophotometer. At this wavelength, only the gold(III) ions absorbed maximally with no interference from other reagents in solution. Under these conditions, the kinetic curves were exponential and rate constants were obtained from plot of log (A_t - A_∞) versus time equation (2)

$$In(At - A_\infty) = In(A_0 - A_\infty) - k_{obs}t \qquad (2)$$

Where A_t is absorbance at time t, A_∞ is absorbance at infinity, A_o is initial absorbance and k_{obs} is pseudo – first order rate constant. Second order rate constants, k_2, were determined as the ratio of k_{obs} : $[C_3H_7OH]$ [9].

The influence of $[H^+]$ and ionic strength ($NaClO_4$) on the rate of the reaction were investigated within the ranges of $[H^+] = (5.0 \times 10^{-4}$ to $1.0 \times 10^{-2})$ mol dm^{-3} and $\mu = (2.0 \times 10^{-2}$ to $1.2 \times 10^{-1})$ mol dm^{-3} respectively while maintaining other reaction conditions constant.

Effect of dielectric constant on rate of reaction

The dielectric constant of the medium was estimated, equation (3) by varying the ratio of acetone to water.

$$D_{reaction\ medium} = \frac{(D_{water} \times V_{water}) + (D_{acetone} \times V_{acetone})}{V_{total}} , \qquad (3)$$

Where D_{water} and $D_{acetone}$ are dielectric constants of water and acetone, V_{water} and $V_{acetone}$ are volumes of water and acetone and V_{total} is the total volume of water when $V_{acetone}$ is zero.

Test for free radical

Exactly 2 g of acrylamide was added to the partially reduced reaction mixture containing various concentrations of oxidant, propanol and hydrogen ion and allowed to stand for about 2 minutes, this was followed by adding a large excess of methanol. The same procedure was repeated for solutions of C_3H_7OH and $AuCl_3(OH)^-$ separately to serve as control. The lack of gel formation was an indication of the absence of free radical during the course of the reaction [10].

Temperature dependence study

At constant $[AuCl_3(OH)]^-$, $[C_3H_7OH]$, $[H^+]$ and ionic strength, temperature was varied between 303 to 318 K using a thermostatic water bath to adjust the reaction temperature. The reagents were quickly mixed after attaining the desired temperature and the reaction was monitored. The temperature of the reaction product was measured at the completion of the reaction to ensure there was no significant loss in initial temperature. Plot of ln (k/T) against 1/T was made equation (4) from where the activation entropy and enthalpy were determined.

$$\ln\frac{k}{T} = 23.759 + \frac{\Delta S^{\mp}}{R} - \frac{\Delta H^{\mp}}{R}.\frac{1}{T} \qquad (4)$$

Product analysis

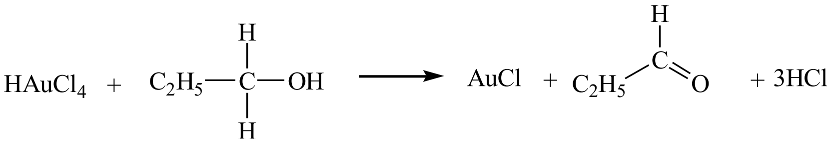
The presence or absence of aldehyde as product from propanol oxidation was tested by combining 2 cm^3 of the reaction mixture with 3 cm^3 of 2, 4-dinitrophenylhydrazine solution. The appearance of yellowish precipitate indicated the presence of aldehyde. A confirmatory test using Fehling's solutions was also done. To 1 cm^3 of Fehling's solution A, equal volume of Fehling's solution B was added until precipitation occurred. About 3 cm^3 of the Fehling's Solution B was added in drops until the precipitate disappeared, then 3 cm^3 of the liquid product was added and boiled. A brick-red colour indicated the presence of aldehyde. Qualitative test as described by [11] with slight modifications was used to check for the absence of Au (III) ion to ensure its reduction. Excess of propanol was reacted with the gold(III) complex ion solution and allowed to proceed to completion. A 2 cm^3 portion of the reaction mixture was afterwards reacted with alkaline solution of hydrogen peroxide as expressed (Equation 5). Lack of precipitate confirmed the absence of Au (III).

$$2[AuCl_4]^- + 3H_2O_2 + 6OH^- \rightarrow 2Au_{(s)} + 3O_{2(g)} + 8Cl^-{}_{(g)} + 6H_2O \quad (5)$$

Results and Discussion

Stoichiometry

Spectrophotometric titration using the mole ratio approach revealed that one mole of $[AuCl_3(OH)]^-$ was reduced by one mole of propanol (Figure 1) and conforms to stoichiometric equation (6).



$$HAuCl_4 + C_2H_5\text{—}CH_2\text{—}OH \longrightarrow AuCl + C_2H_5\text{—}CHO + 3HCl$$

(6)

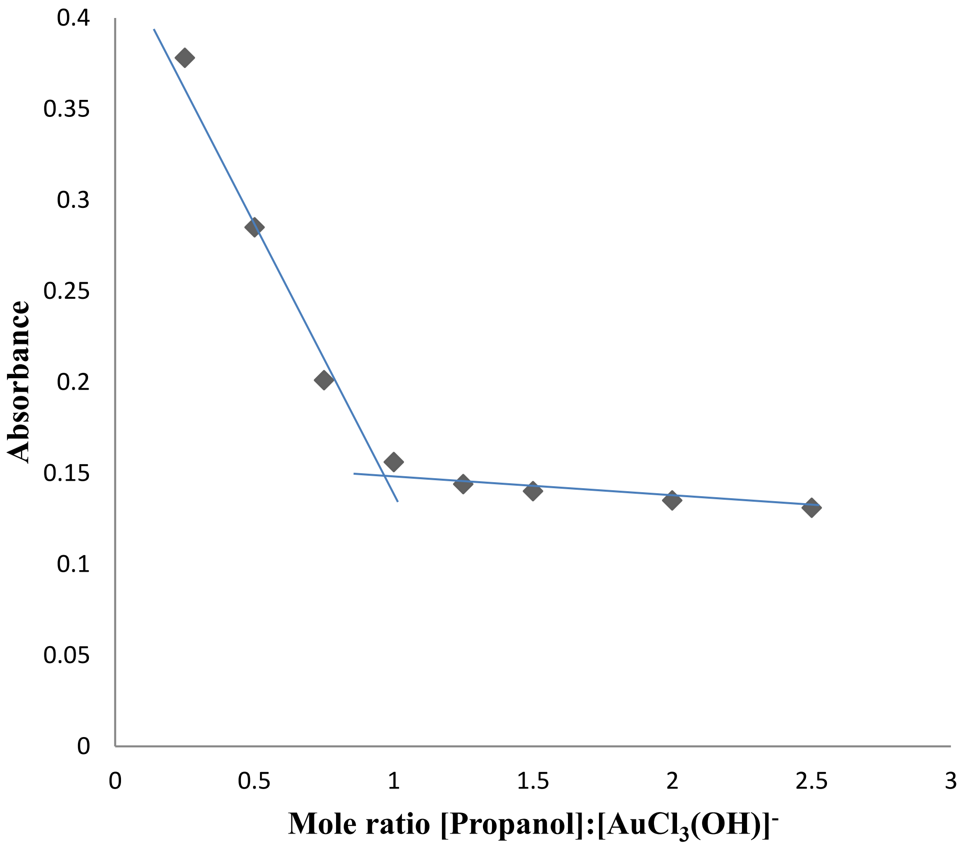
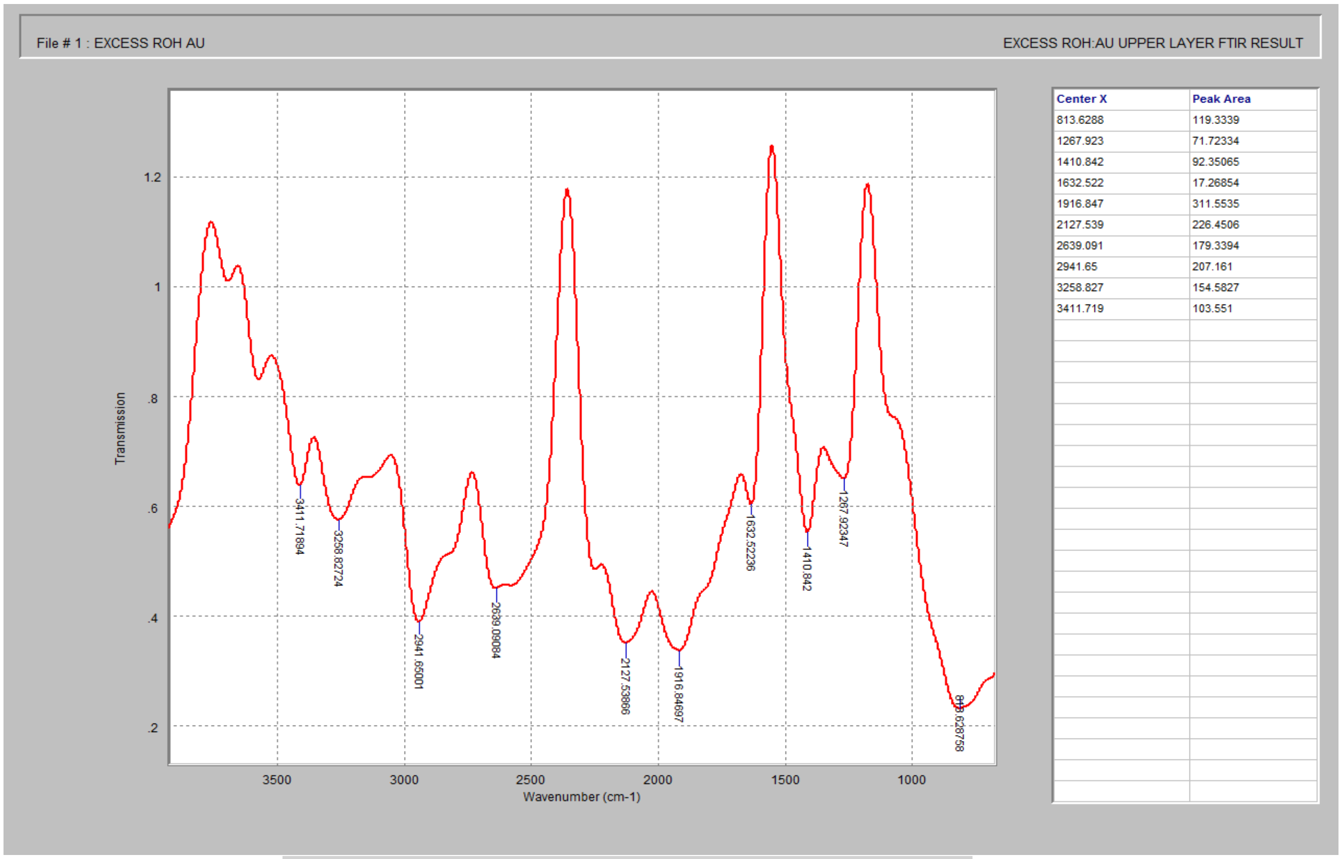


Figure 1: Mole ratio plot for the reduction of $[AuCl_3(OH)]^-$ by propanol

gold(III) complex ion was reduced to gold(I) while propanol was oxidized to propanal. This agrees with findings by [8, 12-13] for the reduction of gold(III) with other substrates. Ether was used to extract the organic product from the reaction and IR spectrum is presented (Figure 2). Band observed at 1762 cm^{-1} confirmed the formation of an aldehyde from propanol oxidation



Center X	Peak Area
813.6288	119.3339
1267.923	71.72334
1410.842	92.35065
1632.522	17.26854
1916.847	311.5535
2127.539	226.4506
2639.091	179.3394
2941.65	207.161
3258.827	154.5827
3411.719	103.551

Figure 2: FTIR spectrum of propanal from propanol oxidation

Kinetics

Pseudo-first order plot of log (A_t -A_∞) against time was linear for up to 85 % of the reaction. The linearity of the plot indicates first-order dependence on the concentration of gold(III). Pseudo first order rate constant, kobs, increased with increase in [C_3H_7OH] (Table 1). Plot of log k_{obs} versus log [C_3H_7OH] was also linear with a slope 0.9825 (Figure 3), indicating a first order with respect to [C_3H_7OH] and a second order overall. The rate equation for this system can be written (Equation 7).

$$\frac{-d[AuCl_3(OH)^-]}{dt} = k_2[AuCl_3(OH)^-][\mathrm{C_3H_7OH}] \qquad (7)$$

Where mean value of k_2 ($dm^3\ mol^{-1}\ s^{-1}$) = 0.8649±0.061. Similar second order kinetics has been reported for the reduction of Au (III) ions with hydroxylamine [14] and [15].

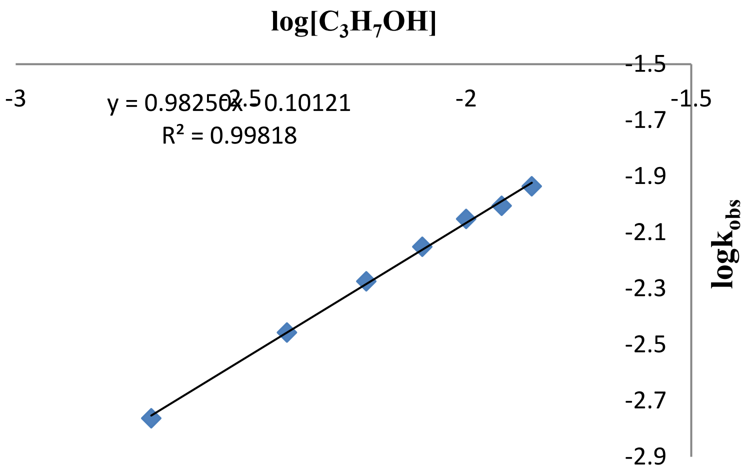


Figure 3: Plot of log k_{obs} against log [C_3H_7OH]

Table 1: Pseudo-first order and second order rate constants for the reaction of [$AuCl_3(OH)]^-$ and C_3H_7OH

$10^3[C_3H_7OH]$, mol dm^{-3}	$10^3[H^+]$, mol dm^{-3}	$10^2\mu$, mol dm^{-3}	10^3k_{obs}, s^{-1}	k_2, dm^3 mol^{-1} s^{-1}
2.0	1.0	5.0	1.73	0.863
4.0	1.0	5.0	3.50	0.875
6.0	1.0	5.0	5.32	0.887
8.0	1.0	5.0	7.07	0.883
10.0	1.0	5.0	8.89	0.889
12.0	1.0	5.0	9.90	0.825
14.0	1.0	5.0	11.63	0.831
6.0	0.5	5.0	5.50	0.940
6.0	2.0	5.0	3.41	0.568
6.0	4.0	5.0	2.16	0.422
6.0	6.0	5.0	1.93	0.368
6.0	8.0	5.0	1.73	0.334
6.0	10.0	5.0	1.57	0.307
6.0	1.0	2.0	5.64	0.940
6.0	1.0	4.0	5.55	0.925
6.0	1.0	5.0	5.37	0.894
6.0	1.0	6.0	5.31	0.868
6.0	1.0	8.0	4.95	0.818
6.0	1.0	10.0	5.11	0.852
6.0	1.0	12.0	5.16	0.860

$[AuCl_3(OH)^-] = 1.0 \times 10^{-4}$ mol dm^{-3}, $\mu = 0.05$ mol dm^{-3} ($NaClO_4$), T = 30 ± 1°C and λ_{max} = 310 nm

Within the acid range $5.0 \times 10^{-4} \leq [H^+] \leq 1.0 \times 10^{-2}$ mol dm^{-3}, the reaction rate decreased with increase in [H^+] (Table 1). Deprotonation of specie before electron transfer mostly result to inverse acid dependence. This kind of rate dependence occurs when a specie exists in forms which are in equilibrium involving H^+ ions with the deprotonated form as the most reactive specie. In this form of dependence, the rate has a limiting value at low [H^+] tending to a zero-value at large [H^+]. In this case, a plot of $(rate)^{-1}$ versus [H^+] would yield a straight line with a non-zero intercept [16]. This suggests that the reaction proceeded by two parallel pathways where one is inverse acid dependent and the other is acid independent as in Figure 4. Hence, the rate as a function of acid can be represented by the general equation (8)

$$Rate = a + b[H^+]^{-1} [AuCl_3OH^-] [C_3H_7OH] \quad (8)$$

Where a = 0.90051 dm^3 mol^{-1} s^{-1} and b = -0.05797 dm^6 mol^{-2} s^{-1}

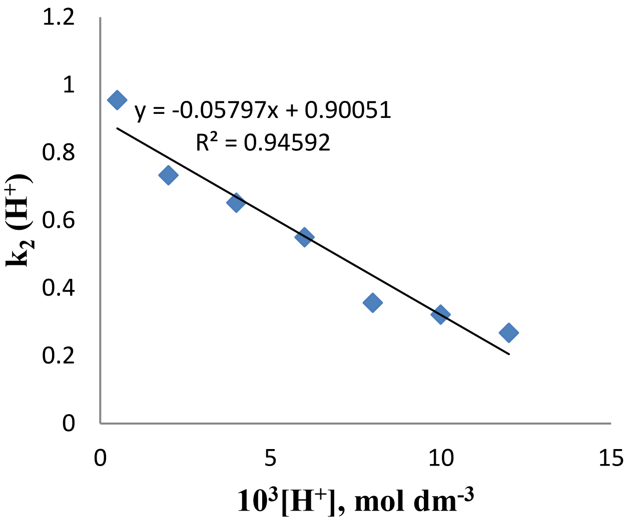


Figure 4: Plot of $k_2(H^+)$ against $[H^+]$

Acid catalysis has also been reported for the oxidation of benzyl alcohols [17-18]. For these reactions acid catalysis was attributed to the protonation of the poor reactive species of these alcohols prior to electron transfer. Alcohols have pKa values from 15 – 20, extension of their alkyl chain as well as branching raises the pKa of these aliphatic alcohols which influences their electron donating ability. Alcohols are strongly basic in nature which makes the OH^- a very poor leaving group and hard to replace. As a result, alcohols are not readily reactive towards substitution and elimination reactions and have to be activated by protonation which converts the OH^- functional group to a weaker base to form positively charged conjugated acid that undergoes substitution and elimination reaction at elevated temperature [19]. An increase in acid concentration in this study should have catalyzed the reaction by forming $PrOH_2^+$ since the reactive form of the oxidant complex is anionic, but the reverse was observed. Inverse acid dependence for this research is therefore attributed to the deprotonation of the oxidant salt in aqueous solution (Equation 9). Increase in hydrogen ion concentration led to common ion effect which retarded the rate of the forward reaction

.

$$HAuCl_4 + H_2O \rightleftharpoons AuCl_3OH^- + Cl^- + 2H^+ \quad (9)$$

Investigating the effect of ionic strength on the rate of the reaction showed that rate was independent of ionic strength concentration, variation in ionic strength concentration had no effect on the rate of the reaction (Table 1). Rate independence on ionic strength means either one or both redox partners is/are neutral [20] or that the reaction occurred between species that formed an ion-pair or adduct [21]. This characteristic is consistent with reactions occurring through outer- sphere mechanism. The non-dependence of rate on ionic strength observed in this reaction is an implication that one of the reactants is not charged at the rate determining step. The absence of primary salt for this reaction is therefore, not unexpected. Similar non-dependence of rate on variation in ionic strength has been reported for the reduction of $AuBr_4$ [15] and $HAuCl_4$ [22]. It was also reported that variation in ionic strength had negligible impact on the rate of methylglycol oxidation by N- bromosuccinimide in alkaline medium catalysed by Os(VII) [23].

Table 2: Effect of medium dielectric constant on the rate of reaction of $[AuCl_3(OH)]^-$ and C_3H_7OH

D	78.40	77.59	76.79	75.99	75.19	74.39	73.59
10^3k_{obs}, s^{-1}	5.48	5.25	5.14	5.37	5.46	5.27	5.34
K2, $dm^3\ mol^{-1}\ s^{-1}$	0.913	0.875	0.856	0.894	0.909	0.879	0.890

$[AuCl_3(OH)^-] = 1.0 \times 10^{-4}$ mol dm^{-3}, $[C_3H_7OH] = 6.0 \times 10^{-3}$ mol dm^{-3}

The effect of medium dielectric constant on the rate of this reaction was investigated by using a binary solvent system of water and acetone from 78.40 to 73.59. Varying dielectric constant had no significant effect on the rate of the reaction (Table 2). This is an indication that the reaction occurred between a charged molecule and a neutral specie, and conforms to the nature of ionic strength dependence observed for this reaction. The result is in conformity with those obtained for the oxidation of oxo-bridged ruthenium (IV) by alcohols [24].

Test for free radical

Addition of acrylamide to the reaction mixture followed by excess methanol did not result to formation of gelatinous precipitate. This showed lack of polymerization of the acrylamide monomers and absence of the participation of free radicals during the reaction. This agrees with findings by [22] for the oxidation of L-tyrosine with gold(III) ion.

Effect of temperature

Effect of temperature on the reaction rate was monitored by altering temperature from 308 to 318 K. Plot of ln(k/T) against T^{-1} was made (Figure 5). The activated enthalpy and entropy evaluated from this plot are 3.668 kJ mol^{-1} and -206.56 JK^{-1} mol^{-1}. The negative value of $\Delta S^{\ddagger}$ obtained is unexpected as it suggests a highly ordered situation. Solvent reorganization can result in negative values of $\Delta S^{\ddagger}$ even for a reaction occurring via an outer-sphere mechanism, hence the assertion that $\Delta S^{\ddagger}$ should be large and negative to indicate an inner-sphere pathway is not completely true [25].

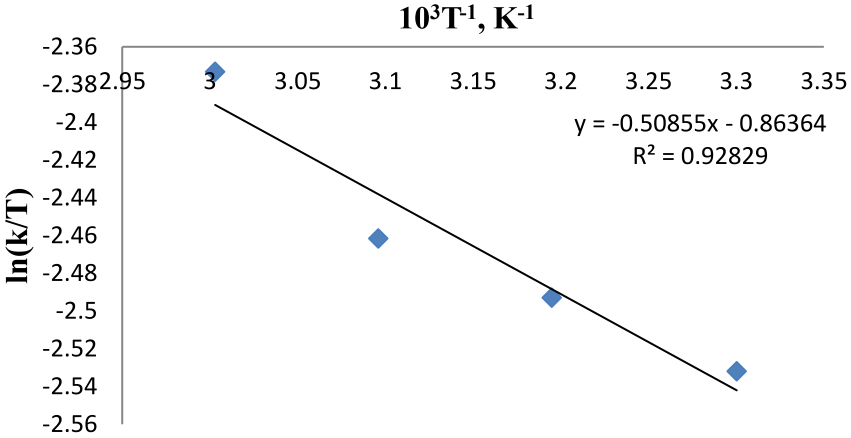


Figure 5: Plot of ln(k/T) vs. T^{-1} for the reduction of $[AuCl_3OH]^-$ by C_3H_7OH

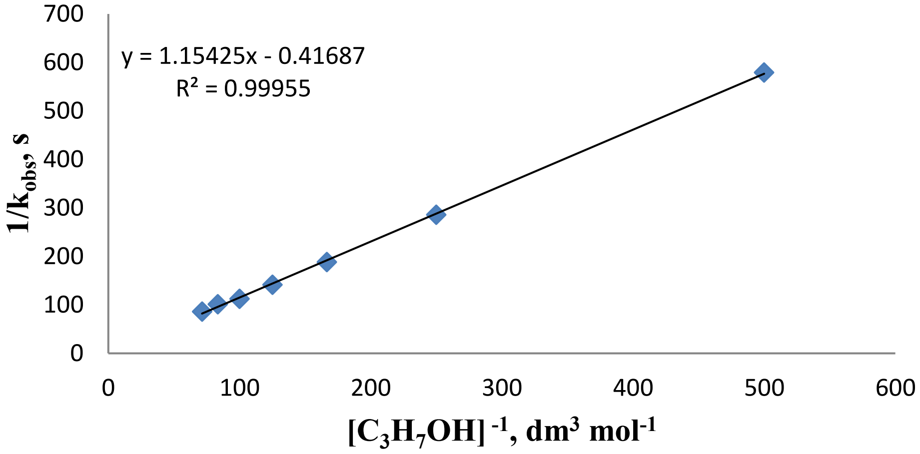


Figure 6: Michaelis-Menten's type plot for the reaction of $[AuCl_3(OH)]^-$ and C_3H_7OH

Michaelis-Menten's type plot of k_{obs}^{-1} versus $[C_3H_7OH]^{-1}$ was made (Figure 6). The plot was linear with negligible intercept which supports the absence of intermediate complex with an appreciable equilibrium constant. Also, comparing the electronic spectrum of $[AuCl_3(OH)]^-$ complex with that of the reaction mixture (Figure 7) showed no shift in λmax of $[AuCl_3(OH)]^-$ at 310 nm, indicating the absence of spectroscopically detectable intermediate complex formation. For enzymatic action where [enzyme] is < [substrate], the rate of formation of product is given by Equation 10

$$d\frac{[product]}{dt} = k_{obs}[E_o] \quad (10)$$

$$k_{obs} = \frac{V\max[S]}{km+[S]} \quad (11)$$

It is observed that taking the reciprocal of Equation 11 and rearranging it, gives Equation 12

$$\frac{1}{k_{obs}} = \frac{1}{V\max} + \frac{km}{V\max}[S]^{-1} \quad (12)$$

For a normal redox reaction, S stands for the reductant. A plot of k_{obs}^{-1} vs. $[S]^{-1}$ gives $V\max^{-1}$ as intercept. If a linear plot which passes through the origin is obtained, it shows that the intercept $V\max^{-1}$ is zero, meaning that Vmax or the equilibrium constant for the active intermediates is zero [26]. The negligible intercept observed for this reaction confirm that the reactions occurred through the outer-sphere mechanism.

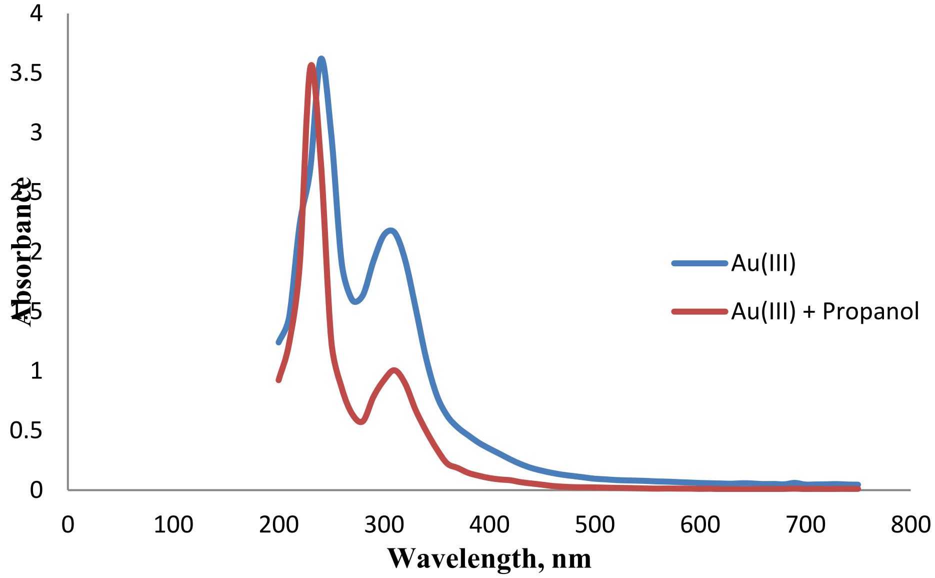


Figure 7: UV-Vis spectrum of reaction mixture after three minutes of reaction

Based on the underlisted evidence obtained from the reaction;

1. Absence of spectroscopically determinable intermediate complex

steps elucidate the experimental data;

2. Negligible intercepts from Michaelis- Menten's type plot

Outer-sphere mechanism is proposed for the reaction; the plausible mechanistic

$$\text{HAuCl}_4 + \text{H}_2\text{O} \xrightleftharpoons{K_1} \text{AuCl}_3(\text{OH})^- + \text{Cl}^- + 2\text{H}^+ \qquad 13$$

$$\text{AuCl}_3(\text{OH})^- + \text{PrOH} \xrightarrow[\text{slow}]{k_1} \text{PrO} + \text{H}_2\text{O} + \text{AuCl}_2 + \text{Cl}^- \qquad 14$$

$$\text{AuCl}_2 \xrightleftharpoons{K_2} \text{AuCl} + 1/2\text{Cl}_2 \qquad 15$$

$$\text{PrOH} + \text{H}^+ \xrightleftharpoons{K_3} \text{PrOH}_2^+ \qquad 16$$

$$\text{HAuCl}_4 + \text{PrOH}_2^+ \xrightarrow[\text{slow}]{k_2} \text{AuCl}_3^- + \text{PrO} + 2\text{H}^+ + \text{HCl} \qquad 17$$

$$\text{AuCl}_3^- + \text{H}^+ \xrightarrow[\text{fast}]{k_3} \text{AuCl}_2 + \text{HCl} \qquad 18$$

$$\text{AuCl}_2 \xrightarrow[\text{fast}]{k_2} \text{AuCl} + 1/2\text{Cl}_2 \qquad 19$$

$$\text{Rate} = k_1[\text{AuCl}_3(\text{OH})^-][\text{ PrOH}] + k_2[\text{PrOH}_2^+][\text{HAuCl}_4] \qquad 20$$

$$\text{Rate} = k_1[\text{AuCl}_3(\text{OH})^-][\text{ PrOH}] + \frac{k_2K_3[\text{PrOH}][\text{HAuCl}_4]}{[\text{H}^+]} \qquad 21$$

$$\text{Rate} = k_1 + \frac{k_2K_3}{[\text{H}^+]}[\text{ PrOH}][\text{HAuCl}_4] \qquad 22$$

Equation 22 is similar to equation 8 where 'a'= k_1 and 'b' = k_2K_3 with values previously stated. This is consistent with the nature of acid dependence observed for this reaction. Aldehyde formation has been reported as the oxidation product of propanol following hydride transfer. Hydride transfer from the alcohol to the oxidant and subsequent formation of the corresponding aldehyde has also been documented for the oxidation of alcohols [27].

Conclusion

The kinetic, thermodynamic and mechanistic studies of the reduction of $[\text{AuCl}_3(\text{OH})]^-$ with propanol were performed. The reaction had a 1:1 mole ratio with respect to oxidant and reductant, unity order with respect to both oxidant and reductant concentrations was observed for the reaction. An inverse acid dependence was noted while changes in ionic strength and dielectric constant of the reaction media had negligible effect on the reaction rate. Activated enthalpy (kJ mol^{-1}) and entropy (JK^{-1}mol^{-1}) for the reaction are 3.668 and -206.56. Polymerization of acrylamide monomers was not observed suggesting the absence of free radicals in the reaction. Michaelis-Menten's type plot had negligible intercept. FTIR spectrum revealed band for C=O resulting from propanol oxidation. Based on the stoichiometry, order of reaction, effect of change in hydrogen ion concentration, medium ionic strength and dielectric constant, test for free radicals, spectrophotometric determination of intermediate complex formation, temperature dependence study, non-conformity of the results with Michaelis-Menten's type plot, outer sphere mechanism has been proposed for this reaction.

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