

## Catalytic Effect of Cetyltrimethylammonium Bromide on the Oxidation of Tetraethylene Glycol by Chloramine-T in Acetic Acid Medium

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The kinetics and mechanism of cetyltrimethylammonium bromide catalyzed oxidation of tetraethylene glycol [2,2'-(oxibis(ethylenoxy)-diethanol)] by chloramine-T in acetic acid medium have been investigated. The reaction has first order dependence on chloramine-T and fractional order for tetraethylene glycol with excess concentration of other reactants. The catalytic effect due to cetyltrimethylammonium bromide has been studied. The small salt effect and increase in the reaction rate with increasing dielectric constant suggest the involvement of neutral molecule in the rate-determining step. Addition of *p*-toluene sulfonamide retards the reaction rate. The effect of chloride ion on the reaction also studied. The effect of temperature on the reaction has been investigated in the temperature range 318-338 K and thermodynamic parameters are calculated from the Arrhenius plot. A tentative mechanism consistent with the experimental results has been proposed.

**Key Words:** Tetraethylene glycol, Cetyltrimethylammonium bromide, Chloramine-T, Oxidation, Kinetics.

### INTRODUCTION

Kinetics is concerned with the rate of the reaction, which influence of various conditions on these rates and mechanisms by which a reaction occurs. The subject of chemical kinetics covers not only reaction rate but also covers a wide range of studies, which includes empirical studies of the effects of the concentrations, temperature, pH, catalyst *etc.* on reactions of various types. It proved to be the best means of study of various reactions such as oxidation, thermal decomposition, halogenation, hydrogenation *etc.* For large-scale production of compounds, it is very essential to know the velocity of the reaction<sup>1</sup>.

Tetraethylene glycol [HO-CH<sub>2</sub>-(CH<sub>2</sub>-O-CH<sub>2</sub>)<sub>3</sub>-CH<sub>2</sub>OH] (TtEG), is a colourless, odourless, non-volatile and hygroscopic liquid. It features a higher boiling point and lower volatility than the lower ethylene glycols. It is completely miscible with water and a wide range of organic solvents, but has only slight affinity for certain

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aliphatic hydrocarbons. TtEG is used as chemical intermediate in manufacture of many ester-based plastics and textiles, in manufacturing of latex paints, surface cleaners, adhesive, hydraulic fluids, printing inks and electronics. TtEG is an excellent choice for automotive engine coolants and heat transfer fluids because of its high boiling point. Oxidation of polyethylene glycols, different alcohols and diols by different oxidant has been carried out by various workers<sup>2-14</sup> but no one has ever used chloramine-T (*p*-Me-C<sub>6</sub>H<sub>4</sub>-SO<sub>2</sub>NCINa.5H<sub>2</sub>O), (CAT) as an oxidant. In continuation of our work<sup>15-17</sup>, cetyltrimethylammonium bromide catalyzed oxidation of TtEG by chloramine-T has been undertaken.

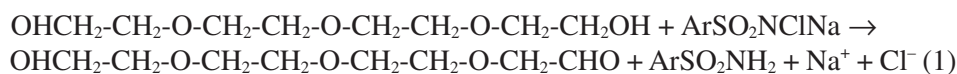
The oxidation of TtEG by acidic chloramine-T was found an extremely slow reaction. The reaction has found to be catalyzed by a cationic surfactant cetyltrimethylammonium bromide (C<sub>16</sub>TAB).

### EXPERIMENTAL

All the chemicals used in this investigation were of AR grade and double distilled water was used for preparation of solutions.

**Rate measurements:** The kinetics and mechanism of cetyltrimethylammonium bromide catalyzed oxidation of tetraethylene glycol was performed titrimetrically. The unused amount of CAT was determined iodometrically<sup>18</sup> at regular time intervals. In all the experiments, the reactions were followed up to two half lives. A constant ionic strength of the reaction mixture was maintained by adding required amount of sodium perchlorate solution.

**Stoichiometry:** Determination of stoichiometry is the stepping-stone for the correct approach towards the mechanistic study of any reaction. Stoichiometry was determined by using varying ratios of the oxidant to TtEG were thermostatted at 25 °C for 48 h incubation and residual CAT was determined iodometrically using standard sodium thiosulphate as titrant and potassium iodide-starch as an indicator. The mole ratio (number of moles of the chloramine-T consumed per mole of TtEG) was calculated. TtEG and chloramine-T react in 1:1 stoichiometry, as follows:



where; Ar = CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>-

**Product analysis:** TtEG (250 mg/250 mL water), perchloric acid (0.1 M/100 mL), C<sub>16</sub>TAB (12 mg/100 mL) and CAT (10 g/100 mL) were mixed for product analysis. After the reaction time over 48 h, the organic components were separated from the mixture into ether; the ethereal extract was dried and concentrated using rotatory evaporator, under low pressure. Using benzene, ethyl acetate mixture 8:2 (v/v) as an eluent, preliminary studies were carried out by thin layer chromatography. A distinct single spot was obtained. A solution of 2,4-DNP was added to it and then the reaction mixture was left overnight at refrigerator temperature (5 °C). The solution was centrifuged and brown-orange residue is obtained. This formation of hydrazine derivative adequately confirmed the oxidation product of the TtEG to be a [2,2'-

oxibis(ethylenoxy)(1-hydroxy)octanal]. The aldehyde was conformed by IR spectra which shows bands at 3445 and 1615  $\text{cm}^{-1}$  for OH stretching and C=O stretching, respectively and a band at 2718  $\text{cm}^{-1}$  for aldehydic C-H stretching. The fact that only one hydroxyl group was attacked and other remained intact gets support from stoichiometry of the reaction as well. Literature survey shows that similar types of oxidative products were reported<sup>15-17,19-21</sup>.

## RESULTS AND DISCUSSION

**Reaction order with respect to chloramine-T:** In a typical kinetic run, for the reaction ( $[\text{CAT}] = 0.004 \text{ mol dm}^{-3}$ ,  $[\text{H}^+] = 0.05 \text{ mol dm}^{-3}$  and  $[\text{TtEG}] = 0.08 \text{ mol dm}^{-3}$ ), a plot of  $\log(a-x)$  versus time (Fig. 1) gave a straight line, which indicates that reaction under the chosen condition follows pseudo first order kinetics. The order with respect to CAT is unity (Table-1). The mean pseudo first-order rate constant,  $k_0$  found to be  $(0.02 \pm 0.2) \times 10^{-4} \text{ s}^{-1}$ .

TABLE-1  
ORDER WITH RESPECT TO CHLORAMINE-T  
 $[\text{TtEG}] = 0.08 \text{ mol dm}^{-3}$ ;  $[\text{H}^+] = 0.05 \text{ mol dm}^{-3}$   
 $[\text{C}_{16}\text{TAB}] = 0.002 \text{ mol dm}^{-3}$ ; Temperature = 328 K

$[\text{CAT}] / 10^{-3} \text{ mol dm}^{-3}$	$k / 10^{-4} \text{ s}^{-1}$ *
0.5	14.46
1.0	6.61
2.0	5.05
3.0	4.21
4.0	2.90
6.0	1.99
8.0	1.13

\*Mean of duplicate experiments

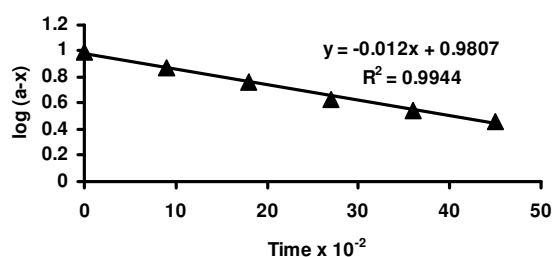


Fig. 1

A slight fall in rate constants with increasing  $[\text{CAT}]$  may be explained on the basis of some deactivation caused by traces of inactive sodium chlorate formed<sup>22</sup> in a side reaction as shown in eqn. 2.



**Reaction order with respect to [TtEG]:** On varying TtEG concentration from 0.02 to 0.24 mol dm<sup>-3</sup>, there is an increase in rate of reaction. The plots of log k *versus* log [TtEG] (Fig. 2, Table-2) gave straight line with slope equal to 0.32 (R<sup>2</sup> = 0.99), suggesting that order with respect to TtEG is fractional. A double reciprocal plot between k<sup>-1</sup> *versus* [TtEG]<sup>-1</sup> (Fig. 3) has been found to be straight line with positive intercept at y-axis. This kinetic evidence of complex formation between the substrate and the oxidant, further support the fractional order dependence.

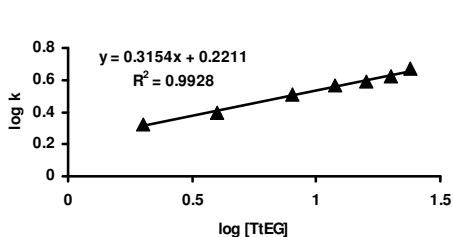


Fig. 2

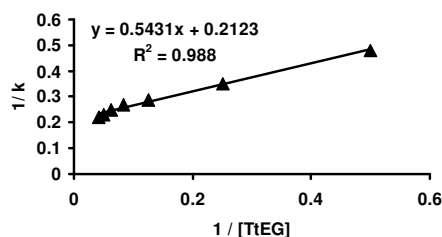


Fig. 3

TABLE-2  
EFFECT OF TETRAETHYLENE GLYCOL CONCENTRATION VARIATION  
[CAT] = 0.004 mol dm<sup>-3</sup>; [H<sup>+</sup>] = 0.05 mole dm<sup>-3</sup>  
[C<sub>16</sub>TAB] = 0.002 mol dm<sup>-3</sup>; Temperature = 328 K

[TtEG] / 10 <sup>-2</sup> mol dm <sup>-3</sup>	k / 10 <sup>-4</sup> s <sup>-1</sup> *
2.0	2.11
4.0	2.49
8.0	3.25
12.0	3.71
16.0	3.89
20.0	4.21
24.0	4.65

\*Mean of duplicate experiments

**Effect of variation of hydrogen ion:** On varying perchloric acid concentration from 0.0125 to 0.2000 mol dm<sup>-3</sup> there is an increase in reaction rate was observed. The plots of log k *versus* log [H<sup>+</sup>] (Fig. 6, Table-3) gave straight line with positive intercept 0.15 (R<sup>2</sup> = 0.98), suggesting that acid plays a complex role in the reaction system. The cause of the increase in the reaction rate with the increasing acid concentration may be attributed to the following equilibria<sup>23</sup>.



It is evident from the above reaction; that an increase in the acid concentration results in the formation of more HOCl, consequently the reaction rate increases.

**Dependence of rate on [C<sub>16</sub>TAB]:** The addition of C<sub>16</sub>TAB in the reaction system catalyzed the reaction. The reaction rate increase with the increase in concentration of C<sub>16</sub>TAB from 0.001 to 0.006 mol dm<sup>-3</sup>. The plot of k *versus* [C<sub>16</sub>TAB]

TABLE-3  
EFFECT OF HYDROGEN ION CONCENTRATION VARIATION  
[TtEG] = 0.08 mol dm<sup>-3</sup>; [CAT] = 0.004 mol dm<sup>-3</sup>  
[C<sub>16</sub>TAB] = 0.002 mol dm<sup>-3</sup>; Temperature = 328 K

[HClO <sub>4</sub> ] / 10 <sup>-2</sup> mol dm <sup>-3</sup>	k / 10 <sup>-4</sup> s <sup>-1</sup> *
1.25	2.44
2.50	2.86
5.00	3.13
10.00	3.32
20.00	3.78
30.00	4.01

\*Mean of duplicate experiments.

(Fig. 5, Table-4) gives a straight line. One of the important properties of micellar systems is their ability to affect the rates of chemical reactions. The reaction rate can either be accelerated or retarded, depending on chemical system, the type and concentration of surfactant and other factors such as pH, ionic strength *etc.* Surfactants are tending to form micelles. The reported critical micellar concentration of C<sub>16</sub>TAB is  $9.2 \times 10^{-4}$  at 25 °C. The effect of surfactants on reaction kinetics is called micellar catalysis<sup>24</sup>. Micelles are formed due to assembling of amphiphilic molecules of surfactant in above known critical micellar concentration<sup>25,26</sup>. Micellar catalysis of reaction in aqueous solutions is usually explained on the basis of a distribution of reactants between water and the micellar 'pseudo phase'. The micelles may provide a favourable orientation of the reactants by polarity gradients. In present case, hydrophobic interaction is most likely to be operative due to relative larger hydrocarbon chains of the substrate<sup>27,28</sup>. This interactive localization of the reacting species in the relatively small volume of the micelles compared to the bulk solution leads to a large increase in the effective concentration and as a result the observed rate increased accordingly. The other most probable reason seems to be electrostatic attraction between polar TtEG molecule and the micelle.

TABLE-4  
EFFECT OF C<sub>16</sub>TAB CONCENTRATION VARIATION  
[TtEG] = 0.08 mol dm<sup>-3</sup>; [CAT] = 0.004 mol dm<sup>-3</sup>  
[HClO<sub>4</sub>] = 0.05 mol dm<sup>-3</sup>; Temperature = 328 K

[C <sub>16</sub> TAB] / 10 <sup>-3</sup> mol dm <sup>-3</sup>	k / 10 <sup>-4</sup> s <sup>-1</sup> *
0.0	0.02
1.0	0.69
2.0	1.19
3.0	1.93
4.0	2.63
4.8	3.27
5.2	3.80
6.0	4.38

\*Mean of duplicate experiments.

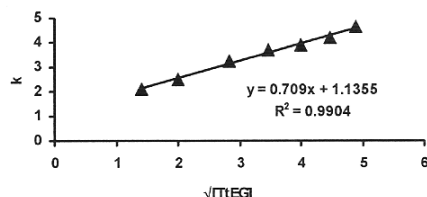


Fig. 4

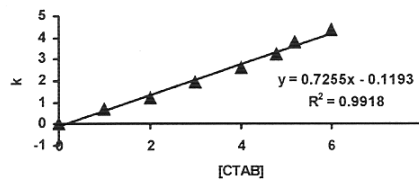
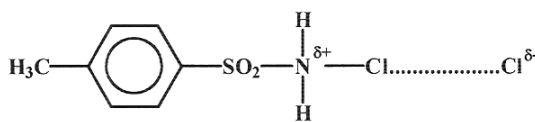


Fig. 5

**Effect of variation of the  $[\text{Cl}^-]$ :** The role of chloride ion in the kinetics and mechanism of the oxidation reaction by CAT is very crucial. On varying chloride ion concentration from 0.002 to 0.030 mol dm<sup>-3</sup>, the rate of reaction increases. The plot of  $\log k$  versus  $\log [\text{Cl}^-]$  (Fig. 7) is a straight line with a slope of 0.12 ( $R^2 = 0.98$ ). The cause of acceleration in the reaction rate is the generation of molecular chlorine/HOCl due to the interaction between a chloride ion and a chloronium ion produced by the hydrolysis of an N-Cl bond, in polar medium. Alternatively, the added chloride ion may coordinate with the protonated oxidant to give rise to species of the type (I).



Type (I)

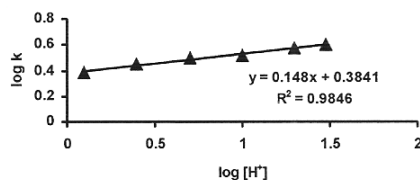


Fig. 6.

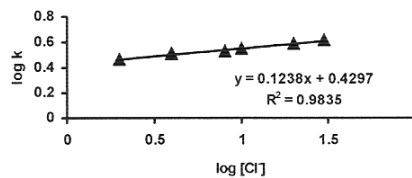


Fig. 7.

**Effect of variation of the ionic strength, dielectric constant and addition of *p*-toluenesulphonamide (PTS):** With the employed reactant concentrations, initial ionic strength of the reaction mixture was 0.058. It was found that there is no substantial change in the reaction rate on varying the ionic strength from 0.058 to 0.118 by the addition of neutral sodium perchlorate. The small salt effect suggests the participation of neutral species in rate determining step *i.e.* the substrate molecule and HOCl. This assumption further got support from effect of change in dielectric constant of the reaction medium. The effect of dielectric constant in reaction medium has been studied by adding acetic acid (40-60 %) in the reaction medium at constant concentrations of other reactants. The rate of reaction increases by increasing the proportion of acetic acid in solvent medium. This supports the involvement of neutral

species in the rate-determining step. Addition of PTS, one of the reaction products, from 0.0005 to 0.0080 mol dm<sup>-3</sup> at constant CAT and TtEG concentration, decreases the rate of reaction. This supports that HOCl is the main oxidizing species.



**Effect of temperature:** By increasing temperature the rate of the reaction increases,  $\log k$  vs.  $1/T$  (Fig. 9) is a straight line in the temperature range 318-338 K. This shows Arrhenius equation is valid for this reaction. From the linear Arrhenius plot activation parameters for overall reaction were evaluated  $E_a = 61.98 \text{ KJ mol}^{-1}$ ,  $\Delta H^* = 59.25 \text{ KJ mol}^{-1}$ ,  $\Delta G^* = 75.20 \text{ KJ mol}^{-1}$ ,  $\Delta S^* = -48.61 \text{ JK}^{-1} \text{ mol}^{-1}$ , respectively.

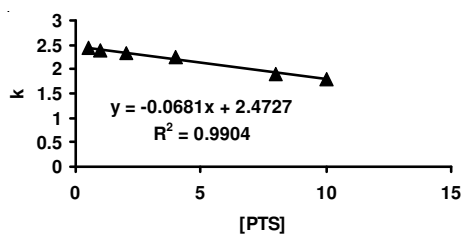


Fig. 8

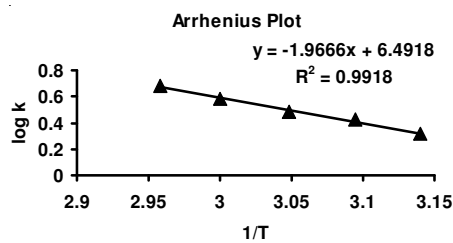


Fig. 9

**Mechanism:** Chloramine-T is a strong electrolyte<sup>23</sup> and in aqueous solution it behaves as follows:



The anion  $\text{ArSO}_2\text{NCl}^-$  is protonated in acidic solutions to give N-chlorotoluene-*p*-sulphonamide.



This  $\text{ArSO}_2\text{NHCl}$  undergoes hydrolysis according to the following reactions:

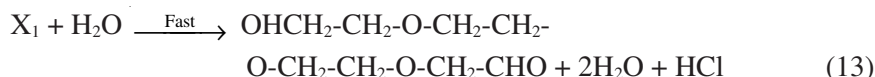
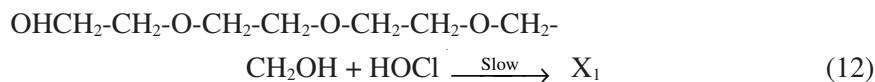


In weakly acidic media (pH = 6-7),  $\text{ArSO}_2\text{NHCl}$  is the prominent reacting species of CAT while at higher acid concentrations (pH < 2.8)  $\text{ArSO}_2\text{NHCl}$  is further protonated according to eqn. 11.



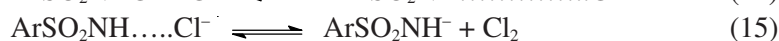
Thus, the possible oxidizing species in acidic solutions of CAT are  $\text{ArSO}_2\text{NH}_2\text{Cl}^+$ ,  $\text{ArSO}_2\text{NHCl}$ ,  $\text{ArSO}_2\text{NCl}_2$  (dichloramine-T) and HOCl. It was found that the reaction rate is retarded on addition of PTS. This is indicative of the fact that either  $\text{ArSO}_2\text{NCl}_2$  or HOCl could be the main oxidizing species. To account for the experimental observations, the following mechanism proposed. The linear double reciprocal plot with intercept on y-axis of  $k^{-1}$  vs.  $[\text{TtEG}]^{-1}$  suggests the formation of complex between the substrate and oxidant.

Thus the rate determining step proposed is:



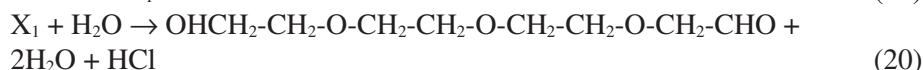
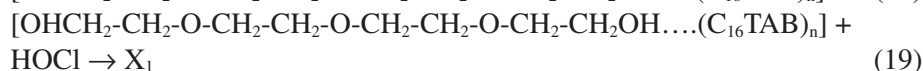
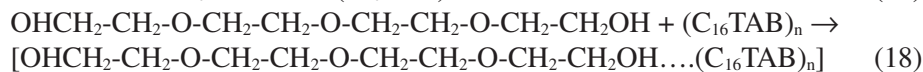
The small salt effect, increase in reaction rate on increasing the dielectric constant and straight line double reciprocal plots between  $1/k$  versus  $1/[\text{TtEG}]$  with positive intercept at y-axis (Fig. 3), support eqn. 12 as being the rate determining step. Thus, the formed complex decomposes quickly into the products, eqn. 13.

**Mechanism in the presence of chloride ions:** On increasing concentration of chloride ions, the reaction rate increases. The order with respect to chloride ions is fractional. The increase in the reaction rate in the presence of chloride ions may be attributed to the following reactions:



Thus, the HOCl generated in eqn. 16 commences oxidizing the substrate simultaneously in a parallel reaction to that in eqn. 12 and 13.

**C<sub>16</sub>TAB catalyzed mechanism:** The reaction between the TtEG and chloramine-T is extremely slow. The addition of C<sub>16</sub>TAB accelerates the reaction substantially. The physical basis for micelle catalysis involves several contributing factors. First there is the effect of the micelle environment on the rate-controlling step in the reaction mechanism. The relative free energies of the reactions and/or the transition state can be altered when the reaction occurs in the micelle phase instead of bulk water. This concept is reminiscent of enzymatic catalysis and many initial studies of micelle systems focused on this possibility. More important consideration is the interactive localization of reacting species in the relatively small volume of the micelle compared to that of the bulk solution. This leads to a large increase in the effectively concentration and the observed rate (in terms of moles per unit time per litre of the entire solution) increases accordingly. The following mechanism is envisioned for the catalysis by C<sub>16</sub>TAB:



**Rate law:** The rate equation for the reaction between TtEG and CAT can be represented by the equation:

$$-d[\text{CAT}] / dt = k_0 [\text{TtEG}]^{1/2} [\text{CAT}] \quad (21)$$

when  $[\text{CAT}]$  in excess above eqn. 21 reduces to



$$r = k'_0 [\text{TtEG}]^{1/2} \quad (22)$$

where the rate constant for catalyzed reaction,  $k'_0 = k_0 [\text{CAT}]$ . The plot between  $k$  and  $\sqrt{[\text{TtEG}]}$  (Fig. 4) gave a linear plot that validates eqn. 22. In the presence of the catalyst, the oxidation proceeds through catalyzed pathways. Therefore, the following represents the rate of depletion of CAT in presence of catalyst under excess [TtEG] and acid concentrations:

$$-d [\text{CAT}] / dt = \{k'_0 + k'_c [\text{C}_{16}\text{TAB}]\} [\text{CAT}] \quad (23)$$

where  $k'_c = k_c [\text{CAT}]$

$$= k'' [\text{CAT}] \text{ where } k'' = \{k'_0 + k'_c [\text{C}_{16}\text{TAB}]\} \quad (24)$$

Equation 24 holds well, when a plot of the observed rate constant in presence of catalyst,  $k$  versus  $[\text{C}_{16}\text{TAB}]$  is linear (Fig. 5).

### Conclusion

The following conclusion can be drawn from the present work under the observed test conditions: (i) The oxidation of the TtEG by acidic chloramine-T was found an extremely slow reaction. The reaction has found to be catalyzed by a cationic surfactant cetyltrimethylammonium bromide ( $\text{C}_{16}\text{TAB}$ ). (ii) The stoichiometry of the reaction between TtEG and CAT is 1:1. (iii) The order with respect to TtEG is fractional and for CAT is one. (iv) The reaction shows increase in rate when hydrogen ion concentration increases. (v) The reaction shows positive chloride ion effect. (vi) The reaction shows positive temperature effect and it obeys Arrhenius relationship. (vii) On the basis of product analysis it is proposed that on oxidation, tetraethylene glycol [2,2'-(oxibis(ethylenoxy)diethanol)] oxidizes in to corresponding aldehyde *i.e.* [2,2'-(oxibis(ethylenoxy)(1-hydroxy)octanal].

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