

Factors influencing Kinetics between potassium peroxodisulphate and potassium iodide by Iodometry

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Available online at: www.isca.in, www.isca.me

Received 22nd February 2018, revised 29th August 2018, accepted 18th September 2018

Abstract

Potassium peroxodisulphate (1) and potassium iodide (2) in a definite concentration mixed together, directly and with taking different catalyst e.g. ferrous sulphate, ferric chloride, sodium sulphate, mercuric acetate and arsenic oxide and rate of reaction is determined by Ostwald isolation method, iodometrically. By varying concentration of (1) and other constant, and vice versa, results suggested that rate of reaction second order; first order with respect to (1) and first order with respect to (2). There is no effect on rate of reaction by adding sodium sulphate, potassium perchlorate while adding mercuric acetate, as increase its concentration, rate of reaction decreases, hence it acts as scavenger. Effect of acids, temperature and addition of compounds on rate of reaction between (1) and (2) has been also studied. Results suggested from experiments that rate of reaction are independent on taking different initial concentration of sulphuric acid as well as perchloric acid. Effect of ferrous sulphate also studied between substrate and oxidant in the presence of sulphuric acid and arsenic oxide (low conc.) and observed that ferrous sulphate acts as inductor, hence the rate of reaction increases as concentration of ferrous sulphate increases. At higher concentration of arsenic trioxide efficacy of inductor is less and As_2O_3 acts as deductor. Arsenic and mercuric salts and sulphate salts cause pollution and affect green environment, directly or indirectly to human health and when related to kinetics As_2O_3 acts as deductor while mercuric acetate act as scavenger.

Keywords: Potassium peroxodisulphate, potassium iodide, iodometry, Ostwald isolation methods, different parameters.

Introduction

Chemical kinetics is a branch of physical chemistry which gives an idea about path of rate of reaction, spontaneity of a reaction, order of reaction and mechanism of a reaction¹⁻³. Various factors which can be affect the rate of reaction like nature and concentration of reactants, temperature, catalysts and surface area of reactants⁴⁻⁶. In view of our research experiments, we have studied kinetics of reaction between oxidant (potassium peroxodisulphate) and substrate (potassium iodide) with addition of different salts, acids and effect of temperature. The different experimental methods used for the determination of the rate law, rate constant and order of reaction. The reaction Peroxodisulphate-iodide; induced by metal ion and metal complexes have been studied under kinetic experiments, such as: rate dependency with respect to oxidant, substrate, inductor, acid, scavenger and other related studies. Here we have been used Ostwald isolation method and progress of reactions carried out iodometrically.

Some salts cause pollution directly or indirectly, affect green environment, human health and in kinetics As_2O_3 acts as deductor while mercuric acetate act as scavenger.

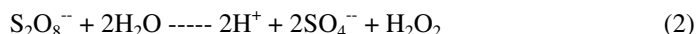
Peroxodisulphate and its Nature: In aqueous solution peroxodisulphate acts as a strong oxidizing agent.

From the heat of formation of peroxodisulphate and the entropy of formation assuming the entropy of the ion to be 35 Cal/deg. The free energy of formation of $H_2S_2O_8$ (or $S_2O_8^{2-}$ since it is a strong acid) is calculated to be -262.0 K.Cal. and the standard electrode potential is given for the reaction⁷⁻⁸.



Being given as -2.01 volts. This Value places peroxodisulphate among the most powerful oxidizing agent. The diacid hydrolysis to give monoacids H_2SO_5 ⁹, called Caro's acid.

The complete hydrolysis of diacid gives hydrogenperoxide.

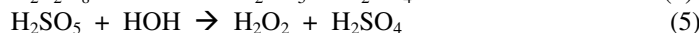
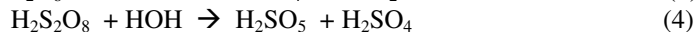
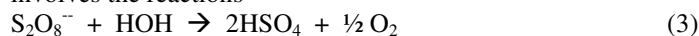


Peroxodisulphate ion is made up of two tetrahedral sulphate groups joined to one another through two oxygen atoms, the O-O link passing through a centre of symmetry. The distance from each S to each of its neighbouring O atoms is $1.50A^\circ$, and that between the two linked O atom is $1.31A^\circ$ (theory S-O 1.70, S=O 1.49, O-O 1.32, O=O 1.10).

Decomposition of Peroxodisulphate: Evidence is given that potassium peroxodisulphate decomposes thermally in aqueous solution¹¹ by two reactions. i. A symmetrical rupture of O-O bond to form two sulphate free radical which disappear by

reaction with water to liberate oxygen. ii. An acid catalysed reaction involving the unsymmetrical rupture of O-O bond.

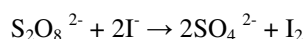
The decomposition of peroxodisulphate in aqueous solutions involves the reactions –



In alkaline, neutral and dilute acid solutions peroxodisulphate decomposes reactions (4) and (5) occur. The Study of potassium and sodium salts of persulfuric acid in alkaline, neutral and dilute acid solutions was first made by Levi and Miglioni later by Green and Masson¹² and by Kailain and Coworkers.

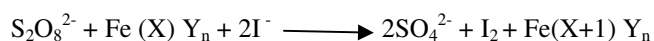
There is general agreement among these workers that persulphate decomposes in aqueous solution is first order. Barthlett and Smith have studied the thermal decomposition of peroxodisulphate in neutral and alkaline medium¹³.

In this experiment the kinetics of the reaction between peroxodisulfate and iodide ions



The liberating iodine is titrated against standard hypo solution using starch as indicator.

Stoichiometry of the reaction: The Stoichiometry of the reaction was studied by taking substrate at lower concentration. For this a known amount of oxidant and substrate was taken in a conical flask, keeping all other factors constant. The reaction mixture was kept for 24 hours for completion of the reaction. After the reaction was over, the estimation of oxidant was done and observed that two moles of substrate is used for one mole of oxidant in the given experimental condition:



Where: X is the valency of inductor and Y is the ligand used for complex inductor.

It has been observed that inductor factor is 2 that are 2 molecule of acceptor is oxidized by one molecule of inductor.

Materials and methods

Chemical required: All chemicals used during kinetic investigations were of either Laboratory Reagent or Analytical Reagent grade. Doubly distilled water was taken for preparing solutions and during kinetic run. Some solutions were covered by black paper in order to protect from the photochemical decomposition.

Oxidant, peroxodisulphate was freshly prepared every time and other solution i.e. Potassium iodide, Sodium sulphate, Potassium perchlorate and Scavenger salts were prepared

according to needed normalities by direct weighing. Acid solutions were also prepared of required normalities.

Procedure: The reaction was carried out at constant temperature 30°C (for other two systems 40°C and 55°C taken) with the help of thermostat. Brown bottles were used to protect from light. Required amount of substrate and catalyst were added. Water was added to maintain the total volume of reaction mixture 50ml.

The reaction was initiated by the addition of known amount of peroxodisulphate solution. The kinetics of reaction was studied by withdrawing aliquot amount (5ml) of reaction mixture at various time intervals. The Result were reproducible with $\pm 5\%$. The order of reaction with respect to peroxodisulphate was determined with the Ostwald isolation method. Following formula was used to determine the rate constant as the reaction in first order.

$$K = 2.303/t \log (a/(a-x));$$

Where: K = First order rate constant, a = Initial concentration of oxidant, (a-x) = the concentration of oxidant at time t.

Factors affecting kinetics: Dependence of Rate on Oxidant

Concentration: In order to understand the effect of (oxidant) on the rate, experiments were carried out at different initial concentrations of potassium Peroxodisulphate and keeping all other factors constant. The results are given in Table-1-2.

Table- 1: Dependence of Rate on Oxidant Concentration.

Tx10 ⁻² Sec.	Vol. of Hypo	Log (a/(a-x))	Kx10 ⁵ Sec ⁻¹
0	0.1	-	-
12	1.45	0.039	7.5
48	4.85	0.1564	7.5
72	6.7	0.2368	7.57
120	9.45	0.3931	7.54
144	10.5	0.4716	7.54
160	11.4	0.5524	7.57
V _∞	15.8	-	-

[K₂S₂O₈] = 2x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, Temp = 303K, Graphical K = 7.44x10⁻⁵S⁻¹, Average K = 7.54x10⁻⁵S⁻¹.

Effect of (Substrate) on Reaction Rate: The effect of (substrate) on the reaction rate has been investigated by conducting reaction at various initial concentrations of Potassium iodide keeping all other factors constant. The results so obtained are summarized in Table-3-4.

Table-2: Dependence of Rate on Oxidant Concentration.

$[S_2O_8] \times 10^3 \text{ mol m}^{-3}$	$K \times 10^5 \text{ Sec}^{-1}$
2	7.54
3	7.55
4	7.54
5	7.52
6	7.54
7	7.56

$[KI] = 2.5 \times 10^{-2} \text{ mol dm}^{-3}$, Temp = 303 K.

Table-3: Effect of Substrate on Reaction Rate

$T \times 10^{-2} \text{ Sec.}$	Vol. of Hypo	$\log(a/(a-x))$	$K \times 10^5 \text{ Sec}^{-1}$
0	0.2	-	-
12	1.55	0.0393	7.54
48	4.95	0.1576	7.56
96	8.25	0.3151	7.56
120	9.5	0.3937	7.56
144	10.55	0.4729	7.56
168	11.4	0.5496	7.53
V_∞	15.8	-	-

$[K_2S_2O_8] = 2 \times 10^{-3} \text{ mol dm}^{-3}$, $[KI] = 2.5 \times 10^{-2} \text{ mol dm}^{-3}$, Temp = 303 K, Graphical $K = 7.51 \times 10^{-5} \text{ S}^{-1}$, Average $K = 7.55 \times 10^{-5} \text{ S}^{-1}$.

Table-4: Effect of Substrate on Reaction Rate.

$[KI] \times 10^2 \text{ mol dm}^{-3}$	$K \times 10^5 \text{ Sec}^{-1}$
1	3.23
1.5	3.72
2	6.25
2.5	7.55
3	8.35
3.5	11.80

$[K_2S_2O_8] = 2 \times 10^{-3} \text{ mol dm}^{-3}$, Temp = 303 K.

Effect of (Sodium Sulphate) on Reaction Rate: In order to understand the effect of [Sodium Sulphate] or effect of addition

of product of the reaction on reaction rate, experiments were carried out at different initial concentration of sodium sulphate, keeping all factors constant. The results so obtained are summarized in Table-5.

Table-5: Effect of Sodium Sulphate on Reaction Rate.

$[Na_2SO_4] \times 10^3 \text{ mol dm}^{-3}$	$K \times 10^5 \text{ Sec}^{-1}$
2	7.15
3	7.2
4	7.36
5	7.44
6	7.38
7	7.45

$[K_2S_2O_8] = 2 \times 10^{-3} \text{ mol dm}^{-3}$, $[KI] = 2.5 \times 10^{-2} \text{ mol dm}^{-3}$, Temp = 303 K.

Effect of [Potassium perchlorate] on the Reaction Rate: In order to observe the effect of Potassium perchlorate or change in ionic strength of reaction solution on the reaction rate, experiments were carried out at different initial concentration of potassium perchlorate, keeping all other factors constant. The results so obtained are summarized in Table-6.

Table-6: Effect of Potassium perchlorate on Reaction Rate.

$[KClO_4] \times 10^3 \text{ mol dm}^{-3}$	$K \times 10^5 \text{ Sec}^{-1}$
2	6.33
3	6.48
4	6.45
5	6.52
6	6.41
7	6.43

$[K_2S_2O_8] = 5 \times 10^{-3} \text{ mol dm}^{-3}$, $[KI] = 2.5 \times 10^{-2} \text{ mol dm}^{-3}$, Temp = 303 K.

Effect of [Mercuric acetate] on the Reaction Rate: For study the effect of scavenger on the reaction rate, reaction was studied at different initial concentration of mercuric acetate, keeping all other factors constant. The results obtained are given in the Tables-7 to 11 and summarized in Table-12.

Effect of $[HClO_4]$ on the Reaction Rate: To determine the dependency of [Perchloric acid] concentration on reaction rate,

experiments were conducted at different initial concentration of perchloric acid, keeping all the other factors constant. The results so obtained are summarized in Table-13.

Effect of [H₂SO₄] on the Reaction Rate: Keeping all other factors constant, experiments were conducted at different initial concentration of sulphuric acid and results so obtained are summarized in Table-14.

Table-7: Effect of Mercuric acetate on Reaction Rate.

Tx10 ⁻² Sec.	Vol. of Hypo	Log (a/(a-x))	Kx10 ⁵ Sec ⁻¹
0	0.2	-	-
12	2.6	0.0281	5.4
48	9.2	0.1127	5.4
96	16.3	0.2269	5.44
120	19.3	0.286	5.48
144	21.6	0.3374	5.4
168	23.9	0.3957	5.42
V _∞	39.9	-	-

[K₂S₂O₈] = 5x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, [Hg(OAc)₂] = 5x10⁻⁴ mol dm⁻³, Temp = 303 K, Graphical K = 5.20 x 10⁻⁵ S⁻¹, Average K = 5.42 x 10⁻⁵ S⁻¹.

Table-8: Effect of Mercuric acetate on Reaction Rate.

Tx10 ⁻² Sec.	Vol. of Hypo	Log (a/(a-x))	Kx10 ⁵ Sec ⁻¹
0	0.1	-	-
12	2.3	0.0247	4.75
48	8.2	0.0991	4.75
96	14.7	0.1991	4.77
120	17.4	0.2485	4.77
144	19.9	0.2999	4.79
168	22	0.3484	4.77
V _∞	39.8	-	-

[K₂S₂O₈] = 5x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, [Hg(OAc)₂] = 1x10⁻³ mol dm⁻³, Temp = 303 K, Graphical K = 4.61 x 10⁻⁵ S⁻¹, Average K = 4.76 x 10⁻⁵ S⁻¹.

Table-9: Effect of Mercuric acetate on Reaction Rate.

Tx10 ⁻² Sec.	Vol. of Hypo	Log (a/(a-x))	Kx10 ⁵ Sec ⁻¹
0	0.1	-	-
12	2.1	0.0223	4.29
48	7.7	0.092	4.41
96	13.8	0.1832	4.4
120	16.5	0.2306	4.42
144	18.6	0.2715	4.34
168	21.1	0.3257	4.45
V _∞	39.9	-	-

[K₂S₂O₈] = 5x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, [Hg(OAc)₂] = 1.5x10⁻³ mol dm⁻³, Temp = 303 K, Graphical K = 4.39 x 10⁻⁵ S⁻¹, Average K = 4.40 x 10⁻⁵ S⁻¹.

Table-10: Effect of Mercuric acetate on Reaction Rate.

T x 10 ⁻² Sec.	Vol. of Hypo	Log (a/(a-x))	K x 10 ⁵ Sec ⁻¹
0	0.1	-	-
12	1.9	0.0201	3.86
48	6.8	0.08	3.85
96	12.4	0.161	3.86
120	14.8	0.2008	3.85
144	17	0.2408	3.85
168	19	0.2807	3.84
V _∞	39.8	-	-

[K₂S₂O₈] = 5x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, [Hg(OAc)₂] = 2x10⁻³ mol dm⁻³, Temp = 303 K, Graphical K = 3.80 x 10⁻⁵ S⁻¹, Average K = 3.85 x 10⁻⁵ S⁻¹.

Effect of temperature on the reaction rate: The reaction was carried out at different temperatures in order to test the validity of Arrhenius equation and to evaluate different kinetic and activation parameters.

A graph is plotted between log K and reciprocal of temperature a straight line is obtained which proves the validity of Arrhenius equation. Results are given in Table 15-18 and summarized in Table-19.

Table-11: Effect of Mercuric acetate on Reaction Rate.

T x 10 ⁻² Sec.	Vol. of Hypo	Log (a/(a-x))	Kx10 ⁵ Sec ⁻¹
0	0.1	-	-
12	1.5	0.0155	2.98
48	5.4	0.062	2.97
96	9.9	0.1227	2.94
120	11.4	0.1511	2.9
168	15.6	0.2142	2.93
240	19.9	0.2988	2.86*
V _∞	39.9	-	-

[K₂S₂O₈] = 5x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, [Hg(OAc)₂] = 2.5x10⁻³ mol dm⁻³, Temp = 303 K, Graphical K = 2.93 x 10⁻⁵ S⁻¹, Average K = 2.94 x 10⁻⁵ S⁻¹.

Table-12: Effect of Mercuric acetate on Reaction Rate.

[Hg(OAc) ₂]x10 ³ mol dm ⁻³	K x 10 ⁵ Sec ⁻¹
0.5	5.43
1	4.76
1.5	4.4
2	3.85
2.5	2.94

[K₂S₂O₈] = 5x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, Temp = 303 K.

Table-13: Effect of perchloric acid on Reaction Rate.

[HClO ₄] x 10 ² mol dm ⁻³	K x 10 ⁵ Sec ⁻¹
1.0	5.64
2.0	5.59
3.0	6.01
4.0	6.11
5.0	6.21
6.0	6.25

[K₂S₂O₈] = 2x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, Temp = 303 K.

Table-14: Effect of Sulphuric acid on Reaction Rate.

[H ₂ SO ₄] x 10 ² mol dm ⁻³	K x 10 ⁵ Sec ⁻¹
0.5	6.13
1	6.22
2	6.31
3	6.66
4	6.71
5	6.82

[K₂S₂O₈] = 2x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, Temp = 303 K.

Table-15: Effect of Temperature on Reaction Rate.

T x 10 ⁻² Sec.	Vol. of hypo	log (a/a-x)	Kx10 ⁵ Sec ⁻¹
0	0.1	-	-
12	1.5	0.0402	7.73
24	2.7	0.078	7.5
36	3.95	0.1212	7.75
48	5	0.1612	7.73
72	6.8	0.2396	7.7
108	9	0.3598	7.7
V _∞	15.9	0.3598	7.7

[K₂S₂O₈] = 2x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, Temp = 303 K, Graphical K = 7.68 x 10⁻⁵ S⁻¹, Average K = 7.70x10⁻⁵ S⁻¹.

Table-16: Effect of temperature on reaction rate.

T x 10 ⁻²	Vol. of hypo	log (a/a-x)	Kx10 ⁴ Sec ⁻¹
0	0.1	-	-
12	1.9	0.0525	1.01
24	3.5	0.1052	1.01
36	5.2	0.1692	1.01
48	6.5	0.2255	1.01
60	7.3	0.641	1.01
72	8.6	0.3353	1.07
V _∞	15.9	-	-

[K₂S₂O₈] = 2x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, Temp = 308 K.

Table-17: Effect of Temperature on Reaction Rate.

T x 10 ⁻² Sec	Vol. of hypo	log (a/a-x)	K x 10 ⁴ Sec ⁻¹
0	0.1		
24	4.9	0.1597	1.53
36	6.8	0.2437	1.55
48	8.3	0.3238	1.55
60	9.5	0.4007	1.53
72	10.5	0.4771	1.53
84	11.4	0.5596	1.53
V _∞	15.7		

[K₂S₂O₈] = 2x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, Temp = 313 K, Graphical K: 1.50x 10⁻⁴ Sec⁻¹, Average K = 1.54 x 10⁻⁴ Sec⁻¹.

Table-18: Effect of Temperature on Reaction Rate.

T x 10 ⁻²	Vol. of hypo	log (a/a-x)	K x 10 ⁴ Sec ⁻¹
0	0.1		
12	3.5	0.1067	2.05
24	6.2	0.2154	2.06
36	8.3	0.3238	2.07
48	9.9	0.4296	2.06
60	11.2	0.5399	2.07
72	12.2	0.6490	2.07
V _∞	15.7		

[K₂S₂O₈] = 2x10⁻³ mol dm⁻³, [KI] = 2.5x10⁻² mol dm⁻³, Temp = 318 K, Graphical K: 2.10 x 10⁻⁴ Sec⁻¹, Average K = 2.06 X 10⁻⁴ Sec⁻¹.

Table-19: Effect of Temperature on Reaction Rate.

Temperature (K)	K x 10 ⁵ Sec ⁻¹
303	7.71
308	10.1
313	15.4
318	20.6

[K₂S₂O₈] = 2X10⁻³ mol dm⁻³, [KI] = 2.5X10⁻² mol dm⁻³.

Effect of Ferrous sulphate on the reaction rate: Experiments were conducted at different initial concentration of ferrous sulphate in order to determine the effect of inductor on the reaction rate in presence of definite concentration of arsenic oxide and results are summarized in Table-1A and 1B.

Table-1A: Effect of ferrous sulphate on reaction rate.

[FeSO ₄] x 10 ⁻⁴ mol dm ⁻³	K x 10 ⁴ Sec ⁻¹
1	4.65
2	5.43
3	7.34
4	11.60

[K₂S₂O₈] = 2x10⁻³ mol dm⁻³, [KI] = 1x10⁻² mol dm⁻³, [As₂O₃] = 4x10⁻⁵ mol dm⁻³, [H₂SO₄] = 8x10⁻⁵ mol dm⁻³, Temp=313 K.

Table-1B: Effect of deductor (Arsenic oxide) on reaction rate.

[FeSO ₄] x 10 ⁴ mol dm ⁻³	K x 10 ⁴ Sec ⁻¹
1	1.81
2	2.40
3	3.55
4	5.42

[K₂S₂O₈] = 2x10⁻³ mol dm⁻³, [KI] = 1x10⁻² mol dm⁻³, [As₂O₃] = 1.2x10⁻⁴ mol dm⁻³, [H₂SO₄] = 8x10⁻⁵ mol dm⁻³, Temp = 313 K.

Results and discussion

Order of reaction with respect to peroxydisulphate: Result from experiments suggested, that values of K obtained are fairly constant Table-2. It is clear that the rate of reaction is independent of initial concentration of peroxydisulphate. And when plotted a graph between log (a-x) and time, parallel lines are obtained. This concludes that the reaction is first order with respect to oxidant.

Order with respect to substrate: It has been observed that the variation in initial concentration of substrate, effects on the reaction velocity. The straight line graph is obtained on plotting log of rate constant versus log of substrate concentrations. The slope value shows first order with respect to substrate concentration. The plot of rate constant and of substrate concentration also gave a linear graph.

Ionic strength and rate constant: The reaction has been carried out at different ionic strengths of reaction mixture and

observed that the effect of variation was negligible. When the reaction was studied at different concentration of adding sodium sulphate, there was no remarkable change was observed. This shows that there may be formation of sulphate radical eg. SO_4^- .

Effect of Scavenger: Reaction was studied at different concentration of mercuric acetate and observed that reaction rate decreases with increases concentration from 0.5×10^{-3} to 2.5×10^{-3} , Table 7-12. It may be due to formation of sulphate radical anion, during the reaction.

Effect of acid on the reaction rate: At different concentration of perchloric acid the rate seems to attain all most constant hence rate constant does not change with change with concentrations of perchloric acid. And also rate is independent on sulphuric acid variation Table-13, 14.

Effect of inductor: The rate of reaction markedly increases with increase in concentration of ferrous sulphate. In presence of sulphuric acid and arsenic trioxide effect of inductor was observed and observed that rate of reaction is accelerated in both systems Table-1A.

Effect of deductor of arsenic trioxide has been observed at higher concentration where efficacy of ferrous sulphate is low observed Table-1B, because rate of reaction was not increased very much as in observed in Table-1A.

Effect of temperature: A graph is plotted between $\log K$ and reciprocal of temperature a straight line is obtained which proves the validity of Arrhenius equation.

Conclusion

Reaction between potassium peroxodisulphate and potassium iodide in presence of various salts affect rate of reactions. The rate of reaction is independent from addition of sodium sulphate and potassium perchlorate while addition of mercuric acetate, rate of reaction decreases. Addition of arsenic trioxide in higher concentration does not increase rate very much compared to low concentration. These salts cause pollution and also retarded rate of reaction (arsenic trioxide, mercuric acetate).

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