# Determination of Silver (I) using the oxidation of O-Di-Anisidine (ODA) with peroxydisulfate – By catalytic Determination

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**Abstract: A Catalytic method for simple and accurate determination of silver is based on the catalytic effect of Silver (I) ion on the oxidation of Ortho Di Anisidine(ODA) by peroxydisulfate is was determined. ODA is used as an indicator in acidic medium. The rate of the decrease in absorbance of ODA (at 450 nm) is proportional to the concentration of silver in the range of 0.3-4.0 µg/ mL. The theoretical limit of detection was 0.25 µg/ mL. The method is free from most interference. The method was applied to the determination of silver in Lake Water By using different temperatures pseudo-first order rate constants are measured as a function of catalyst concentration. The LOD and relative standard deviation for Silver(I) were 45 mol.dm-3 and 0.6% respectively. The coefficient of variance and % error were in the range of 0.907- 0.238, 0.1-0.30% respectively. River and other water samples from the Samalkota, East Godavari district of Andhra Pradesh state-INDIA were carried out for analysis by using ODA.**

***Key words: Silver (I), ODA, Catalytic Determination, peroxydisulfate.***

## I. INTRODUCTION

P. R. Bontchev[1] et.al., proposed a method for the oxidation of sulphanilic acid with persulphate, catalysed by traces of silver(I). The un-catalysed kinetic equations of the and catalytic reactions, influence of the acidity, the temperature and the activator concentration are carried out. In view of that catalytic method was proposed to determine 1–35 ng of $Ag^+$ per ml with 2.3 percentage relative error. Few metal ions along with silver was also presented. Safavi A[2] et.al., proposed a sensitive and selective determination of silver based on the catalytic effect of silver(I) ion on the oxidation of Janus Green by peroxodisulfate was developed. In this reaction activator is o-Phenanthroline. The rate of the decrease in absorbance of Janus Green found to be 615 nm which is proportional to concentration of silver in the range of 0.3-4.0 ng mL(-1). The theoretical LOD was identified as 0.25 ng mL(-1). The method was applied to the determination of the uptake of silver by plants, lake water, photographic solutions, green plants and other variety of samples. A.M. Afonso[3] et.al., described a method for the determination of silver by using kinetic fluorimetric method. The catalytic effect on the oxidation of pyrocatechol-1-aldehyde 2-pyridylhydrazone by peroxodisulphate is found. In aquatic solution strength of the silver 0.2–0.8 µgml can be determined, and as a stabilizer 10–80 ngml in the presence of 1,10-phenanthroline. From the oxidation the samples obtained are $\lambda_{ex}$ 357 nm and $\lambda_{em}$ 445 nm. Ali A. Ensafi[4]et.al., presented Silver has a very strong catalytic effect on the peroxodisulphate oxidation of Brilliant Cresyl Blue in the presence of 1,10-Phenanthroline as an activator. The reaction is monitored by using UV- spectrophotometer for determining the rate of change in absorbance of brilliant cresyl blue at 635 nm at a temperature of 30°C. Ag(I) is identified at 0.3 – 1500.0 ng/ml. The LOD is 0.1 ng/ml of Ag(I). Silver in samples were successfully carried out. Qin Wei[5] et.al., proposed a catalytic method for the determination of trace metal of Ag+ by using Triton X-100 in micellar medium. The method is based on the catalytic effect of Ag(I) on the rate of a ligand substitution reaction between potassium ferrocyanide and 4,7-biphenyl-1,10-phenanthroline. By using the induction period reaction was followed at a wave length of 536 nm. The Beer's law was in the range of 0–0.040 µg/mL molar absorptivity is 2.43 × 106 L mol−1 cm−1. The reaction influence on variables and interference of foreign ions were reported. The


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proposed method has been applied to the wastewater samples with satisfactory results. Percentage recovery was between 93.2 and 107% and the RSD was less than 4.0%. Kinetic catalytic method for the determination of Ag is also presented [6] -[10]

## II. APPARATUS

Perkin-Elmer Model 35- Spectrophotometer containing a 1-cm glass cell was used for absorbance measurements trace quantity of Ag(I) by using fixed wavelength at 329. By using Schimedzu, UV-Spectrophotometer Absorption spectra was recorded by using 1-cm glass cell. To place the reaction 300C thermostat bath made with Gallenkamp Griffin, BJL-240 V is used. For recoding reaction time of spectra stopwatch was used.

## III. EXPERIMENT

**Reagents:** The chemicals used in this estimation were analytical grade and triply distilled water is used during the experiment. Sample substances are purchased E-Merck Pvt.Ltd.,

***PREPARATION OF SOLUTIONS:***

**ODA:**

The ODA Sample was collected from the Merck specialities pvt.Ltd. The requisite quantity of ODA sample was weighed and diluted and prepared in the 250 ml volumetric flask.

***PREPARATION OF SILVER SAMPLE SOLUTION:***

Silver solution in 500ml was prepared by dissolving 0.07875g AgNO3 in water and diluted with triply distilled water and marked upto the 10ml volumetric flask.

***PREPARATION OF PDS:***

0.250M Potassium peroxodisulphate solution was prepared by dissolving 4.224 g K2S208 in a 250 ml volumetric flask and diluted to the mark with triply distilled water.

***PREPARATION OF 1,10-PHENANTHROLINE SOLUTION:***

1,l0-phenanthroline solution (6.5 x l0-2 mol.dm-3)was prepared by dissolving the compound in 100 ml ethanol in a 100 ml volumetric flask.

## IV. RECOMMENDED METHOD

All the solutions were preheated to the working temperature of 30 ± 1OC in a thermostat bath. A sample solution or silver containing less than 1.00 mol.dm-3of Ag(I) was transferred into a 10ml volumetric flask. Then 1.0 ml O.10 M H2S04 solution, 1.0 ml of 3.25mol.dm-3, 10-phenanthroline and 1.0 ml of 1.89 x 10-2mol.dm-3 ODA were added to the flask. Then water was added to make the solution volume up to mark. 10.0ml of 0.25 mol.dm-3 K2S208 was added into the solution and the solution was diluted to the 50.0ml with water. After the addition of adding the water the time is noted at the end of final addition of solution. A portion of the reaction mixture was transferred within 20 sec. The absorbance was noted at 624nm. By using fixed time method i.e.,0.5-3.0min from beginning of the reaction. Measurements are repeated for silver to get values for uncatalyzed reaction. Net reaction rate was calculated by using change in the absorbance of catalyzed and unanalyzed determination. For large amounts of silver (1.0 - 15.0 pg Ag(I)), 1.0 ml of 0.10 mol.dm-3 H2S04, 1.0 ml of 3.78 x 10-2 mol.dm-3 ODA, 1.0 ml of 3.25 x 10-2 mol.dm-3 1,lO-phenanthroline was used to construct a calibration graph.

## V. CONSTRUCTION OF CALIBRATION CURVE

A straight line was obtained by drawing the graph between Δx Vs Ag(I) concentration. The straight line so observed passing through the origin (Fig-1) this is treated as a calibration curve. Keeping the strength as constant and by using various strengths of Ag(I) Six Δx determinations are calculated. To estimate the standard deviation variance, percentage error and calibration curve Δx measurement is continued for six times and the strength of Ag(I) is measured in each reaction shown in Table 1.

***Determination Of Silver In River Water and Panchromatic Plates***

The proposed reaction is developed by analyzing Samples of Waste and river water which are collected from the Samalkota, East Godavari district of Andhra Pradesh state-INDIA were carried out for analysis by using ODA. The results are compared with AAS. By using ODA solution of 10µg-1 the feasibility of the reaction is identified. The results so measured by using ODA and by using the PDS is presented in Table 1. For untreated waste water from the industry Fruit full results are obtained. Metal ions should not be separated in initial stages. In Contiunation to the above method we are developed a panchromatic plate, about 2 .05 g of the plate is treated with 20 ml of 2M NaOH. This is continued till the try acetyl cellulose membrane HNO3 is added. The Ag(I) Solution is filtered and the filtrate was made up to volume in a 100 ml volumetric flask with water. The suitable aliquot of the solution was analyzed by the proposed method.

## VI. INTERFERENCES

To study the effect of different categories of anions and cations the rate of reaction at PH= 3.4 – 3.6 was measured by using 0.010 µg/ml of Ag(1) was determined(Tab1e 2). The


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tolerance values are tabulated in the table: 2. various foreign ions are not interfered in this reaction. The tolerance limit of Lithium, sodium, potassium ammonium, Barium, periodate, zinc, Iron, Mercury, copper, nickel, cyanide etc.,

## VII. RESULTS AND DISCUSSION

ODA is a reagent that can be oxidized by oxidizing agents such as PDS. On the other hand, the oxidation of ODA by PDS is increased in the presence of ultratrace amounts of silver, and 1,lO-phenanthroline as an activator. This reaction can be followed spectrophotometrically by monitoring the change in absorbance at 420 nm. Figure 1 shows the absorption spectra of ODA at different times. The effect of some N-donor substances previously employed for activation of Ag(1)-catalyzed oxidation reaction was examined, and their efficiency was found to decrease in order, 1, 10-phenanthroline, N-aminopyridine, 2,2-bipyridine, and ammonia. Thus 1,l0-phenanthroline was used as an activator to give greater sensitivity.

## VIII. CONCLUSION

An inexpensive and seletive method is proposed for the determination of Ag (I) by kinetic spectrophotometric technique. First order kinetic reaction by using ODA is followed in this method. The rate equation follows linear relation with concentrations of PDS ion catalyst Silver and [H+]. This proposed method is applied to determine the Ag (I) in different water samples collected from different areas of coastal Andhra by using standard addition method.

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## Figures and Tables

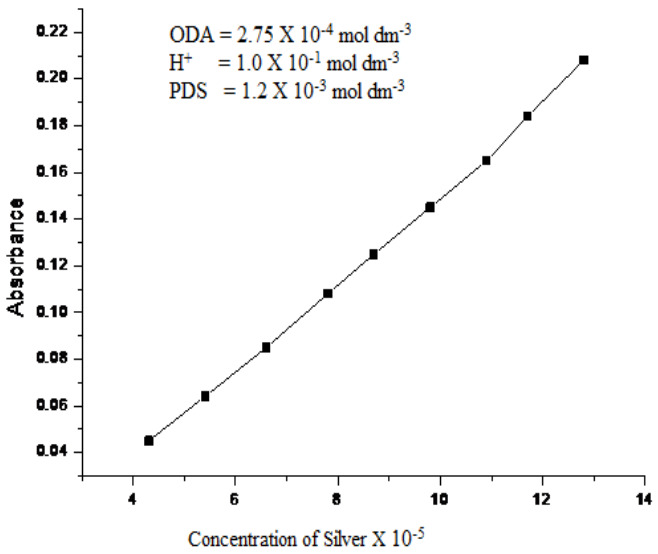


**Fig.1. Calibration graph for the kinetic catalytic determination of Silver.[ODA] = 2.75× 10-4 mol.dm-3, [PDS] = 1.2×10-3 mol.dm-3, [H+] = 1.0 x 10-1 mol.dm-3 μ = 0.1, Temperature = 30± 1 ºC.**

**Table-1 Determination of Ag(I) in waste Water (5.0gr/sample)**

| S. No | Area of the waste water | Metal ion found | | Recovery % present method |
|---|---|---|---|---|
| | | Present method* (μg) | AAS method(μg) | |
| 1 | Samalkota | 4.6 | 4.5 | 97.8 |
| 2 | Bikkavolu | 3.8 | 3.9 | 97.6 |
| 3 | Raja Nagaram | 2.7 | 2.8 | 97.4 |

**[ODA] = 2.75× 10-4 mol.dm-3, [PDS] = 1.2×10-3 mol/dm-3, [H+] = 1.0 x 10-1 mol.dm-3 μ = 0.1, Temperature = 30± 0.1 ºC.**

**Table : 2 Tolerance limits of foreign ions, Tolerance ratio [Species(X)]/[Silver]**

| Species x | Tolerance ratio x/ Silver | Species x | Tolerance ratio x/ Silver |
|---|---|---|---|
| Lithium | 1000 | Perodate | 500 |
| Sodium | 1000 | Zinc | 500 |
| Potassium | 1000 | Iron | 500 |
| ammonium | 1000 | Mercury | 200 |
| Barium | 1000 | copper | 200 |
| Strontium | 1000 | Nickel | 200 |
| palladium | 1000 | Cyanide | 800 |
| Thorium | 1000 | Titanium | 800 |
| cesium | 1000 | Chromium | 800 |
| barium | 1000 | Thiosulphate | 800 |
| Calcium | 1000 | Ferrous | 800 |

**[ODA] = 2.75× 10-4 mol.dm-3, [PDS] = 1.2×10-3 mol/dm-3,[H+] = 1.0 x 101 mol.dm-3 μ = 0.1, Temperature = 30± 1 ºC.**

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