

ESTIMATION OF CHLORINE IN WATER SAMPLES-ELECTROANALYSIS

Shobharani Panchagnula

Department of chemistry, G.Narayanamma Institute of Technology and Science (Affiliated to INTU-H), Telangana, India.

***_____

Abstract - A comprehensive and detailed investigation of chlorine analysis was done by electro analytical and volumetric techniques. In the present study various water samples were tested for the presence of chlorine. Water samples were collected from different depths and investigated by potentiometric and volumetric studies.Both the methods used for analysis were found to be simple and less time consuming, whilst electro analytical technique was found to be more accurate and the results were found to be reproducible.

Key Words: Electro analytical studies, volumetry, potentiometry.

1. INTRODUCTION

There is a great speculation and concern about the effect of chlorination upon organic and inorganic materials in waste and natural waters.

Free chlorine, HOCl are widely used for drinking water sterilization , cooling- water treatment and cyanide treatment(1).

Chemical disinfectants are used to improve the quality of water. The most common disinfectant is chlorine because it is inexpensive and effectively destroys a large number of water borne pathogens. The U.S. Environmental Protection Agency (EPA) enforces regulations regarding the minimum amount of chlorine which must be present in drinking water(2). The Safe Water Drinking Act of (SWDAA) state that a chlorine residual (typically the allowable minimum is 0.2 mg/l) must be present at the points of water consumption(3). But, chlorine reacts with certain organic compounds to produce tri halothanes (THM's), suspected to be carcinogenic (4). Again, the U.S. EPA regulates the maximum amount of THM's which can be present in a DWDS (0.1 mg/l) [5]. The water quality control problem can then be viewed in terms of regulating chlorine levels within a prescribed set of bounds: a lower limit set to ensure that disinfection is taking place, and an upper limit set to minimize harmful side effects. Chlorinated water typically enters the system from a number of sources and is transported to users through multiple paths of pipes, associated with travel time through the pipes. Due to these time delays there is a difficulty in meeting the control objectives because chlorine levels decay over time as water disinfection takes place(6).

Chloride which is present in water combines with calcium, magnesium or sodium forming salts. Chloride occurring in ground water, sea water and also the run off from road can make its entry into water resources(7).

Low levels of chloride are harmless whilst high concentrations of chloride can damage plants and also give unpleasant taste to drinking water and corrode the plumbing, appliances and water heaters due to which toxic metals leach into water (8, 9).

1.1 METHODOLOGY:

The amount of chlorine in different water samples was analysed by volumetric and potentiometic analyses.

Volumetric analysis:

Ground water samples from various depths were analysed for the presence of chlorine. The water samples were first treated with acid and later with potassium iodide and the liberated iodine was measured by iodometric titration. The water samples on acidification were further treated with KI, wherein iodine was liberated. The liberated iodine is treated with hypo solution in the presence of starch indicator. The disappearance of the blue colour is the end point of the titration.

Samples of standard potassium dichromate were prepared by 0.64 g of dichromate salt in 100 ml the standard flask and is made upto the mark.

To 20 ml of different water samples, taken in the conical flask . 20 ml of KI is added. The solution was acidified with HCl in order to create an acid medium. The flask was covered and kept in dark for few minutes to avoid any photo chemical reaction. The liberated iodine was now titrated against hypo in the presence of starch indicator until the colour changed from blue to green and thus the dichromate was standardized. The green colour is due to the chromium ion. Different water samples were now titrated volumetrically against standard hypo solution. Samples were acidified with glacial acetic acid, potassium iodide added, kept in dark for five minutes and titrated further with hypo using starch as indicator.

Potentiometry

Determination of chlorine in waters, with sequential detection by potentiometric sensors, is done. The equipment

Т



used consisted of a potentiometer (a potential measuring device), a reference electrode and an indicator electrode (a chloride ion selective electrode). The half cell potential of the reference electrode is a known constant and this electrode is completely insensitive to the composition of the solution under study. A series of standards containing 10 – 100 micro grams per litre of chlorine are prepared. A chloride ionselective electrode Orion 93-07 was used to check the analytical signal. The electrode potential was measured by an Orion pH/mVmeter 407 A to 1 mV. For calibration standard solutions of 10 -1 to 10-4M sodium chloride were used. As a known addition reagent 10-2 M sodium chloride solution was used. For direct potentiometry a standard graph was used. The one-step known addition was performed .Six known addition (0.10, 0.15, 0.20, 0.25 and 0.30 ml of 10.2 M sodium chloride) were added to 10 ml of the sample and after each addition the electrode potential was checked and recorded. It is worth mentioning that the analysis was performed with constant stirring.

2. RESULTS AND DISCUSSION:

Titrimetry:

Standardisation of hypo:

TABLE-1

S.No.	Vol. dichromate	of	Buret readir Initial Final	te ng	Volume hypo down	e of run
1	20		0	3.2	3.2	

Hypo was standardized using standard potassium dichromate solution (0.022M). The results are tabulated as shown in Table -1 and the calculations are as shown.

<u>K₂Cr₂O₇</u>	Hypo		
M ₁ =0.022	M ₂ =?		
V ₁ = 3.2	$V_2 = 20$		
n ₁ = 1	n ₂ =6		
$M_2 = (M_1V_1/n_1)^*(n_2/V_2) = 0.021$			

Estimation of chloride:

The results for the estimation of chloride in water samples are shown in Table-2 and the calculations shown.

TABLE-2

S.No.	Vol. of different	Burette reading		Volume	of
	water samples	Initial		hypo	run
		Final		down	
1	20	0	8.8	8.8	

Instrumental Analysis:

Estimation of chloride

TABLE-3

S.No.	$\Delta E/\Delta V$	Volume of hypo
1	2	0
2	3	1
3	4	2
4	4	3
5	28	4
6	19	5
7	5	6
8	2	7
9	2	8
10	3	9
11	5	10
12	3	11
13	2	12
14	2	13

The results for the estimation of chloride by potentiometry are shown in Table-3 and the graph is as shown in figure-1

Graph of $\Delta E/\Delta V$ Vs. Volume of hypo in potentiometry







3. CONCLUSIONS

An attempt to develop a simple and accurate method for chloride analysis was done in the present study. Chloride analysis was done volumetrically and potentiometrically .The results were found to be reproducible and accurate (10,11).

The analysis by potentiometric in the present study was found to be more accurate than volumetric analysis. Potentiometric titrations are based on standard electrode potential change observed through potentiometer, whilst direct titrations are based on physical observation on color change. The detection of the endpoint can be noted significantly by a drastic change in potential (in potentiometry) .So both precision and accuracy could be achieved. But in the case of direct titration using an indicator, change in the color is the criteria and the observation of color change can vary from one person to other and so both precision as well as accuracy cannot be achieved. For instance using starch indicator, the color change observation may significantly vary from person to person. Electroanalytical analyses eliminate any indicator blank error and pinpoint accuracy over volumetric titration was obtained (12, 13). Hence it could be concluded that electroanalytical techniques are better methods for analysing chloride ions in water.

ACKNOWLEDGEMENT

The author is thankful to the Chairman, Joint secretary and Principal of G.Narayanamma Institute of Technology and Science for providing all the facilities to carry on the research work.

REFERENCES

1.Determination of Chlorine Dioxide, Chlorine, Chlorite, and Chlorate in Water.E. Marco Aieta, Paul V. Roberts and Margarita Hernandez. Journal (American Water Works Association)Vol. 76, No. 1, Water Storage: Problems and Solutions (JANUARY 1984), pp. 64-70.

2.Chlorine in Water by the Use of Diethyl-p- The Determination of Free and Combined phenylene Diamine .Arthur T .PalinJournal (American Water Works Association) Vol. 49, No. 7 (July 1957), pp. 873-880.

3.Determination of octanol/water partition coefficients for hydrophobic organic chemicals with the "slowstirring" method. Jack De Bruijn*, rans Busser, Willem Seinen andJoop Hermens. Version of Record online: 20 OCT 2009.DOI: 10.1002/etc.5620080607.

4. E. Marco Aieta, Paul V. Roberts and Margarita HernandezJournal (American Water Works

Association)Vol. 76, No. 1, Water Storage: Problems and Solutions (JANUARY 1984), pp. 64-70.

5. The Determination of Free and Combined Chlorine in Water by the Use of Diethyl-p-phenylene Diamine Arthur T. PalinJournal (American Water Works Association)Vol. 49, No. 7 (JULY 1957), pp. 873-880.

6 .Biodegradation of cis –dichloroethene and vinyl chloride in the capillary fringe Zohre Kurt, E. Erin Mack, and Jim C. Spain Environmental Science & Technology **2014** *48* (22), 13350-13357.

7.The occurrence eof organohalides in chlorinated waters.T.A. Bellar, J.J. Lichtenberg and R.C. Kroner Journal (American Water Works Association)Vol. 66, No. 12, Disinfection (December 1974), pp. 703-706.

8.Determination of Trace Amounts of Chlorine in NaphthaJ G Bergmann and John Sanik Anal. Chem., 1957, 29 (2), pp 241–243

9.The Determination of Free and Combined Chlorine in Water by the Use of Diethyl-p-phenylene Diamine Arthur T. Palin Journal (American Water Works Association)

10.Measurement of Henry's law constants for C1 and C2 chlorinated hydrocarbons James M. Gossett Environ. Sci. Technol., 1987, 21 (2), pp 202–208.

11.Methods for the photometric determination of reactive bromine and chlorine species with ABTS. Elieiver vol.34, Issue 18 December 2000, Pages 4343-4350

12. Determination of free and total potential haloforms in drinking water Arnold A. Nicholson, Otto. Meresz. And Brenda Lemyk Chem., 1977, 49 (6), pp 814–819.

13.Use of syringaldazine in a photometric method for estimating "free" chlorine in water Robert. Bauer and Chauncey O. Rupe Anal. Chem., 1971, 43 (3), pp 421–425.

Т