**LABORATORY REPORT**

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| ESTIMATION OF “AVAILABLE CHLORINE” IN BLEACHING POWDER |
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| **AIMS/OBJECTIVES:**   1. Learn and determine the amount or fraction of available chlorine in bleaching powder. 2. Determine the colour changes during the estimation of available chlorine in the bleaching powder. 3. Determine the chemical reactions that occur in the chlorine determination. |
| **INTRODUCTION/THEORY:**  Bleaching powder, white or nearly white powder that is usually a mixture of calcium chloride hypochlorite, CaCl (OCl); calcium hypochlorite, Ca (OCl) 2; and calcium chloride, CaCl2. Sometimes called chloride of lime, it can be prepared by reacting calcium hydroxide or slaked lime, Ca (OH) 2, with chlorine gas, Cl2. It is used as a strong bleaching agent, as a disinfectant, and in making Javelle water. Bleaching powder was first produced in 1799 by Charles Tennant in Glasgow, Scotland. (<http://www.answers.com/main/topictitle=bleaching+powder&method=2&tname=bleaching-powder>)  Calcium hypochlorite is a chemical compound with formula Ca (ClO) 2. It is widely used for water treatment and as a bleaching agent (bleaching powder). This chemical is considered to be relatively stable and has greater available chlorine than sodium hypochlorite (liquid bleach).  It is manufactured using the calcium process or the sodium process.  Calcium Process  2 Ca(OH)2 + 2 Cl2 → Ca(ClO)2 + CaCl2 + 2 H2O  Sodium Process  2 Ca(OH)2 + 3 Cl2 + 2 NaOH → Ca(ClO)2 + CaCl2 + 2 H2O + 2 NaCl  Bleaching powder is actually a mixture of calcium hypochlorite Ca(ClO)2 and the basic chloride CaCl2, Ca(OH)2, H2O with some slaked lime, Ca(OH)2. (http://chemicalland21.com/industrialchem/inorganic/CALCIUM%20HYPOCHLORITE.htm")  Chlorine plays an important biological role in the human body, where the chloride ion is the principal anion in intracellular and extracellular fluids. Large quantities of chlorine are used to produce insecticides, such as DDT Chlorine is widely used as an industrial bleaching agent of paper and textiles. Ordinary household laundry bleach contains the active ingredient sodium hypochlorite (about 5 percent of masses), which is prepared by reacting chlorine gas with a cold solution of sodium hydroxide:  Cl2(g) + NaOH(aq) -------- NaCl(aq) + NaClO(aq) + H2O(l)    Cl2 is liberated upon the treatment of bleaching powder with HCl  OCl-(aq) + Cl-(aq) + 2H+(aq) --------- Cl2(aq) + H2O(l)  The available chlorine refers to the chlorine liberated by the action of the dilute acids on the hypochlorite and is expressed as a percentage by weight in the case of bleaching powder. Commercial bleaching powder contains 36-38 percent of available chlorine.  Two methods are in common use for the determination of the available chlorine. In the first, the hypochlorite solution or the suspension is treated with an excess of a solution of potassium iodide, and strongly acidified with acetic acid:  OCl- + 2l- + 2H+ ------------ Cl- + l2 + H2O  The liberated iodine is titrated with standard sodium thiosulphate solution. The solution should not be strongly acidified with hydrochloric acid, for the little calcium chlorate which is usually present, by virtue of the decomposition of the hypochlorite, will react slowly and react with potassium iodide and liberate iodine:  ClO3- + 6l- + 6H+ ------- Cl- + 3l2 + 3H2O  In the second method, the hypochlorite solution or suspension is titrated against standard arsenite solution; this is best done by adding the excess of the arsenite solution and then back titrating with the standard iodine solution.  In this experiment, the first method would be used. Available chlorine in bleaching powder would be determined by treating a suspension of the powder with KI and H2SO4. The liberated chlorine will set free from the KI an equivalent amount of iodine which is titrated against a standard thiosulphate solution. |
| **CHEMICALS & EQUIPMENT:**   1. Bleaching powder 2. Beaker 3. Distilled water 4. Burette 5. Standard ‘hypo’ solution 6. Turbid solution of bleaching powder 7. Pipette 8. 250ml conical flask 9. 1% starch solution 10. KI solution 11. H2SO4 12. 250ml volumetric flask. |
| **PROCEEDURE:**   |  |  | | --- | --- | |  |  | | TEST | OBSERVATION | | -2.5g of bleaching powder was weighed and transferred into a beaker. |  | |  |  | | -The bleaching powder was turned into paste using little amount of distilled water and was transferred into the 250ml volumetric flask. |  | |  |  | | -The volumetric flask was filled up to the mark with distilled water and was shaken well. | A milky/chalky solution of a turbid solution of bleaching powder was obtained. | |  |  | | -The burette was rinsed and filled with a standard hypo solution to the zero mark. |  | |  |  | | -50ml of the turbid solution was pipette into a 250ml conical flask and 2g of KI and 15ml of H2SO4 was added to the solution and was well shaken. | Initial yellowish orange colour was obtained with the addition of KI.  On the addition of H2SO4, a dark brown colour was observed. | |  |  | | -The solution was then titrated with the standard hypo solution | Light yellowish colour was obtained | |  |  | | 2ml of 1% starch solution was added to the solution and again titrated with the remaining hypo solution. | Deep blue colour was obtained initially. Further titration resulted in a colorless solution. | |  |  | | The titration was repeated two more times to obtain some more concordant values. |  | |  |  | |
| **CALCULATIONS:**  **Titration table**   |  |  |  |  | | --- | --- | --- | --- | | Burette readings/cm3 | 1 | 2 | 3 | | Initial volume | 0.00 | 0.00 | 0.00 | | Final volume | 24.00 | 24.00 | 23.80 | | Titre value | 24.00 | 24.00 | 23.80 | |
| Average titre = 24.00 + 24.00 + 23.80 = **23.93cm3**  3  The equations representing the experiment are:   1. Ca(OCl)Cl + 2HCl ------- CaCl2 + H2O + Cl2 2. Cl2 + 2KI ---- KCl + I2 3. I2 + 2Na2S2O8 -------- Na2S4O6 + 2NaI   **For the first titration**  [Na2S2O3] = 0.50M  V(Na2S2O3) = 24.00cm3  n(Na2S2O3) = c[Na2S2O3] x v(Na2S2O3)  = 0.50 x 24.00  1000  =12 x 10-3mol  n(I2) = x n(Na2S2O3)  = 6 x 10-3mol  but n(I2) = n(Cl2)  ∴ n(Cl2) = 6 x 10-3mol  This amount is contained in 50cm3 of the turbid solution.  ∴ The amount of Cl2 in the 250cm3 turbid solution is  = 250 x 6 x 10-3  50  = 0.03mol  ⇒m (Cl2) = n(Cl2) x M(Cl2)  = 0.03 x 71  = ***2.13g***  **For the second titration**  [Na2S2O3] = 0.50M  V(Na2S2O3) = 24.00cm3  n(Na2S2O3) = c[Na2S2O3] x v(Na2S2O3)  = 0.50 x 24.00  1000  =12 x 10-3mol  n(I2) = x n(Na2S2O3)  = 6 x 10-3mol  but n(I2) = n(Cl2)  ∴ n(Cl2) = 6 x 10-3mol  This amount is contained in 50cm3 of the turbid solution.  ∴ The amount of Cl2 in the 250cm3 turbid solution is  = 250 x 6 x 10-3  50  = 0.03mol  ⇒m (Cl2) = n(Cl2) x M(Cl2)  = 0.03 x 71  = ***2.13g***  **For the third titration**  [Na2S2O3] = 0.50M  V(Na2S2O3) = 23.8cm3  n(Na2S2O3) = c[Na2S2O3] x v(Na2S2O3)  = 0.50 x 23.80  1000  =11.9 x 10-3mol  n(I2) = x n(Na2S2O3)  = 5.95 x 10-3mol  but n(I2) = n(Cl2)  ∴ n(Cl2) =5.95 x 10-3mol  This amount is contained in 50cm3 of the turbid solution.  ∴ The amount of Cl2 in the 250cm3 turbid solution is  = 250 x 5.95 x 10-3  50  = 0.0298mol  ⇒m (Cl2) = n (Cl2) x M(Cl2)  = 0.0298 x 71  = ***2.11g***  The mean mass X = 2.13 + 2.13 + 2.11  3  = **2.123g**   |  |  |  |  | | --- | --- | --- | --- | | X/g | X mean | (X-X)/g | (X-X)2/g2 | | 2.13 | 2.123g | 0.007 | 4.90 x 10-5 | | 2.13 | 2.123g | 0.007 | 4.90 x 10-5 | | 2.11 | 2.123g | -0.013 | 1.69 x 10-4 |   = 4.90 x 10-5 +4.90 x 10-5 +1.69 x 10-4 =***2.67 x 10-4*** g2  Standard deviation (S) =  Where n = 3 and f = 1  =  = **0.00943g**  Relative standard deviation =  =  = **0.44%**  Percentage of Cl2 in sample =  mt = mass of sample  =  = **84.92%**    b) Normality is the number of equivalents of a defined species per litre according to the specified reaction. In this definition the number of hydrogen referred to may be replaced by the equivalent amount of electricity, or by one equivalent of any other substance, but the reaction to which the definition is applied must be clearly specified.  Normality = Number of equivalents  Number of L  Number of mill-equivalents  Number of mL  c) The concentration of 4N H2SO4 in moldm-3 is 4moldm-3 because  Concentration = Number of moles (equivalents)  Number of dm-3 (L)  Since 1L = 1000dm-3  **DISCUSSION:**  When the bleaching powder was reacted with the sulphuric acid, the chlorine obtained was in the gaseous form and so potassium iodide was immediately added to the product of the reaction to prevent the gaseous chlorine from escaping. This is because it formed a salt when reacted with the potassium iodide. But in this state, the chlorine available cannot be determined so the turbid solution is titrated against hypo solution. This enabled us determine the number of moles of iodide which in turn enable us to determine the available chlorine in the sample.  The nature of starch indicator to easily combine with iodine to form complex species was the reason why it was not used at the start of the titration. That is why the titration was allowed to proceed to certain point before the starch indicator was added.  The standard deviation of the data collected was found to be equal to **0.00943g**. This value is far less than 1; hence it implies the average mass of chlorine in the sample calculated was very close to the true mass. The magnitude of the relative (percentage) deviation determined shows that the calculated mass only deviated from the actual mass by a percentage of **0.44%.**  The percentage mass of chlorine in the bleaching powder is **84.92%** which implies that more than half of the bleaching powder constituted chlorine. |
| Normality is the number of equivalents of a defined species per litre according to the specified reaction. In this definition the number of hydrogen referred to may be replaced by the equivalent amount of electricity, or by one equivalent of any other substance, but the reaction to which the definition is applied must be clearly specified.  The concentration of 4N H2SO4 in moldm-3 is 4moldm-3 .  **ERROR ANALYSIS:**   1. A few drops of the standard hypo solution might have dropped out due to the slightly faulty burette which was used. |
| **PRECAUTIONS:**   * The equipment were washed before and after each experiment to prevent impurities from contaminating the reagents. * The burette readings were taken from below the meniscus in order to prevent wrong readings through parallax errors. * During the titration process the standard hypo solution was added drop by drop in order to ensure that the reaction does not largely exceed its end point. * All mixtures were well shaken to ensure a homogenous distribution of the molecules of the solutions. * A bulb or pump was used to pipette the turbid solution due to its corrosive ability. |
| **CONCLUSION:**  The magnitude of the relative (percentage) deviation which was determined showed that the calculated mass only deviated from the actual mass by a percentage of **0.44%** hence, it can be concluded that the average mass of chlorine in the sample calculated was very close to the true mass. The percentage mass of chlorine in the bleaching powder was about more than half the content of bleaching powder. This indicated that chlorine forms a great portion of the bleaching powder. |
| **REFRENCES:**   1. ***The Columbia Electronic Encyclopedia***, Sixth Edition, Copyright © 2003, Columbia University Press. Licensed from Columbia University Press. All rights reserved. [www.cc.columbia.edu/cu/cup/](http://www.answercentral.com/main/Record2?a=NR&url=http://www.cc.columbia.edu/cu/cup/) 2. <http://www.answers.com/topic/bleaching-powder/> 3. <http://chemicalland21.com/> 4. Medical Dictionary, the American Heritage® Stedman's Medical Dictionary, Copyright © 2002, 2001, 1995 by Houghton Mifflin Company. |