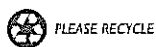




A Division of Phibro Animal Health Corporation

Analysis of Alk-Cu-Strip Solutions for Copper, Chlorite, and Chloride



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Summary:

Copper can be determined by an EDTA titration. In the absence of copper, chlorite can be determined by a thiosulfate titration. If copper and chlorite are both present, the thiosulfate titration will produce the sum of the two. In this case, the copper concentration, if significant, must be subtracted from the sum to get the chlorite concentration.

You may not ever need to determine the chloride concentration.

Apparatus Needed:

| | |
|-------------------------|----------|
| 25 ml buret | Stirrer |
| 250 ml Erlenmeyer flask | pH meter |

Chemicals Needed:

- 0.15N Sodium Thiosulfate Solution ($\text{Na}_2\text{S}_2\text{O}_3$)
- 1% Starch Solution
- Potassium Iodide (KI) Solid
- Deionized or Distilled Water (DI Water)
- Acetic Acid (CH_3COOH)
- 1.0 N Hydrochloric Acid (HCl)
- Methyl Orange Indicator

Procedure:

For Alk-Cu-Strip Part A:

1. Pipet 2.00 ml solution into an Erlenmeyer flask, add DI water to about 100 ml, titrate with 1.00 N HCl using methyl orange indicator.

Calculations:

$$\text{Alkalinity (N)} = \frac{N(\text{HCl}) \times \text{ml}(\text{HCl})}{\text{ml solution}}$$

For Alk-Cu-Strip Part B (no copper):

1. Prepare a working solution by diluting part B 1 to 20. Put 10.0 ml into a 200 ml volumetric flask and add DI water to the mark.
2. Pipet 5.0 ml of working solution into a 250 ml Erlenmeyer flask. Add DI water to a volume of 75-100 ml. Acidify with 10 ml glacial acetic acid.
3. Add 2-3 g potassium iodide (KI).
4. Titrate the released iodine with thiosulfate (S_2O_3) solution to a clear-colorless endpoint. The endpoint may fade in and out for a few minutes. Continue adding thiosulfate until the solution remains clear.

Calculations:

$$\text{NaClO}_2 (\text{g/l}) = [N(\text{S}_2\text{O}_3) \times \text{ml}(\text{S}_2\text{O}_3)] 90.5$$

$$\% \text{NaClO}_2 = \frac{[N(\text{S}_2\text{O}_3) \times \text{ml}(\text{S}_2\text{O}_3)] 9.05}{\text{Density Part B Soln. (g/l)}}$$

$$\text{NaClO}_2 (\text{ppm}) = 10,000 \text{ NaClO}_2 (\%)$$

For Solutions Containing Appreciable Amounts of Copper (i.e., Bath Solutions):

1. Pipet 1.00 ml solution into a 250 ml Erlenmeyer flask. Add DI water to a volume of 75-100 ml. Acidify with 10 ml glacial acetic acid.
2. Add 2-3 g potassium iodide (KI).
3. Titrate the released iodine with thiosulfate (S₂O₃) solution to a clear-colorless endpoint. If the endpoint fades in and out continue adding thiosulfate until the solution remains clear.
4. Determine the copper concentration using a Hach kit.

Calculations:

$$NaClO_2(g/l) = \left[\frac{N(S_2O_3) \times ml(S_2O_3)}{ml \text{ solution used}} - \frac{Cu(g/l)}{63.5} \right] 22.6$$

$$NaClO_2(\%) = \left[\frac{N(S_2O_3) \times ml(S_2O_3)}{ml \text{ solution used}} - \frac{Cu(g/l)}{63.5} \right] \frac{2.26}{\text{Density soln. (g/ml)}}$$

$$NaClO_2(ppm) = 10,000 NaClO_2(\%)$$

To Determine Chloride in Chlorite Containing Solutions (Cu will not interfere):

For NaClO₂, at about 25%:

1. Pipet 1.0 ml solution into a 250 ml Erlenmeyer flask, add 100 ml DI water, add 1g sodium bisulfite, mix, then add a little 50% nitric acid to a pH of 0.3.
2. Add about 4 drops phenosafranin, then titrate with 0.2N silver nitrate solution. About 17 ml silver nitrate solution will be needed. The color change should be from pink to blue. The precipitate will be colored, not the solution. A magnetic stirrer should be used because the color change may be hard to see.

Calculations:

$$\text{Total Chloride(g/l)} = \frac{N(\text{AgNO}_3) \times \text{ml}(\text{AgNO}_3)(35.5)}{\text{ml sample used}}$$

The sodium bisulfite produces chloride from NaClO_2 . So, this chloride must be subtracted from the total chloride. There should not be an appreciable amount of chloride in either part A or part B. The bath may contain small amounts of chloride. To titrate a solution from the bath use 4.00 ml solution and all other quantities the same as above.

This is the chloride of interest.

$$\text{Chloride not from } \text{NaClO}_2 \text{ (g/l)} = \text{Total Chloride (g/l)} - (0.392) \text{ NaClO}_2 \text{ (g/l)}$$

The last term, the NaClO_2 term, comes from a thiosulfate titration.

Phenosafranin may be purchased from Fluka (1-800-358-5287). One (1) gram will last a long time. To make the indicator solution, dissolve 0.1g phenosafranin in 100 ml distilled or deionized water.