The Bleach Strength Test — A Chemical Test Method to Determine the Strength of Sodium Hypochlorite

Background

The liquid sodium hypochlorite made by the Powell Continuous Bleach Manufacturing Plant or by other methods is produced as sodium hypochlorite (NaOCl) in the presence of excess caustic. Because consumers require differing solution concentrations, bleach manufacturers need to measure specific components of the product. Sodium hypochlorite (NaOCl), excess caustic (NaOH), and excess alkalinity (Na₂CO₃) are components in bleach that are routinely tested. Although this test procedure does not address heavy metals and sodium chlorate (NaClO₃), a decomposition product, they should be tested at intermittent intervals and will require a qualified laboratory to perform these tests.

Comparative Sample Analysis

The test method will provide accurate and repeatable results by the producer and consumer to ensure the product is meeting the desired specification.

However, there are times when the strength of the sodium hypochlorite as tested by the manufacturer will differ from the consumer’s results. Many times these differences can be accounted for by the losses in shipping due to decomposition. However, it is difficult to know when the differences in test results are decomposition losses and when they are errors in test methods or procedures.

In order for both the manufacturer and consumer to verify testing methods and decomposition losses, an easy procedure can be used to verify the accuracy and repeatability of each other’s testing method and the decomposition losses that result due to shipping time and temperature.

At the time of loading, the manufacturer will take two retained 250 ml samples of the sodium hypochlorite loaded in the tank truck or shipping container. The manufacturer will place each sample in a separate insulated container with ice of sufficient quantity to keep the samples cold.

The manufacturer will retain one iced sample and send one iced sample with the shipment to be given to the consumer for testing. At the time of delivery, the consumer will take one more 250 ml retained sample of the sodium hypochlorite unloaded from the tank truck and place this third sample in another insulated container with ice.

Within a few hours of the arrival of the samples to the consumer’s laboratory, the consumer will notify the manufacturer that the testing of the iced shipped samples will commence and both the manufacturer and the consumer will test each iced sample a total of three times. The consumer will also test the iced delivered sodium hypochlorite from the tank truck three times. By comparing results, both the manufacturer and the consumer will have data on the repeatability and accuracy of each other’s results and the decomposition of the product during shipping will have been established for that given delivery time and temperature.

The differences in the results between the consumer and the manufacturer of the sodium hypochlorite should be a 0.5% or less. For example, if a test resulted in a strength of 13.70% by weight, a 0.5% error would mean the sample could have been from 13.63 to 13.77%. When comparing strength of bleach between the producer and consumer, this error can be significant. If these results are not achieved, a comparative review of laboratory methods and equipment may be required.

Analytical Method

The analytical methods described in this application note require that certain glassware and laboratory apparatus be available for use. The methods can be easily performed using pipettes and volumetric glassware. The level of accuracy (and cost savings on chemicals) is directly related to the measurement. For example, higher degrees of accuracy will provide better quality control of the product and can potentially reduce manufacturing costs of the product. The following laboratory apparatus with their Fisher Scientific catalog numbers are presented to help you equip your facility with the minimum laboratory glassware and accessories needed to analyze your manufactured bleach.
Laboratory Apparatus
Erlenmeyer flask (250 ml) 10-041A (pack of 6)
Volumetric Pipette (5 ml) 13-650-2F (pack of 6)
Volumetric pipette (10 ml) 13-650-2L (pack of 6)
Volumetric pipette (25 ml) 13-650-2P (min of 6)
Volumetric pipette (50 ml) 13-650-2S (min of 6)
Pipette blub 14-070-1 (pack of 3)
Liquid Dispensers (optional) 5-30 ml 13-706-18 (optional)
Weighing bottle 03-422F (pack of 6)
Volumetric flask (250 ml) 1-205D (min of 6)
Graduated cylinder (10 ml) 08-555A (min of 3)
Graduated cylinder (50 ml) 08-555C (min of 3)
Burette (50 ml) 03-700-12B (min of 2)
Burette support 14-688 (min of 1)
Magnetic stirrer 14-493-120S (min of 1)
Magnetic stir bar 14-511-62 (min of 10)
Analytical balance (0.001 g) 01-920-76 (min of 1)
Reference weight (30 g) 01-920-76 (min of 1)
Hydrometer (1.000 - 1.050) 11-603-7A
Hydrometer (1.050 - 1.100) 11-603-7B
Hydrometer (1.100 - 1.150) 11-603-7C
Hydrometer (1.150 - 1.200) 11-603-7D
Hydrometer (1.200 - 1.250) 11-603-7E
Hydrometer (1.250 - 1.300) 11-603-7F

Suggestions for Setting up Analytical Procedures
• Obtain a representative sample by flushing the sampling port so that the sample is fresh and free of foreign matter and then slowly take the sample over 30-60 seconds.
• Sample should be stored in a cool, dark place and analyzed as soon as possible. NOTE: If the sample cannot be tested immediately, the sample should be stored with ice or in a 40°F (5°C) refrigerator.
• Use good pipetting technique. Use a pipette bulb to draw solution into the pipette to a point above the level mark. Allow the bottom of the meniscus to reach the level mark. When dispensing the sample into a flask, touch the tip of the pipette to the side of the flask to remove only the “hanging drop”.
• Before titrating the sample, rinse the titration burette with acid, distilled water, and finally with the titrant. Fill the burette. Open the stopcock to allow the titrant to descend to the 0.00 ml mark. Make sure that the burette tip does not contain an air bubble.
• Make sure you swirl or thoroughly mix the titrant and sample in the flask during the titration.
• Refill the burette for the next titration.

High Accuracy Analytical Methods
• Tare a weighing bottle on the analytical balance. Pipette 25 ml of the bleach sample into the weighing bottle and weigh to the nearest 1.0 mg (0.001g). This is the weight of the original sample:

\[
\text{Specific gravity of the bleach} = \frac{\text{sample weight}}{25 \text{ ml}}
\]

• Transfer the sample to a 250 ml volumetric flask, washing all of the sample out of the weighing bottle into the volumetric flask with distilled water. Dilute to the mark with distilled water and mix thoroughly. Aliquots of this sample will be used for the sodium hypochlorite, excess caustic, and excess sodium carbonate titrations. Typical size aliquot solutions are 5 or 10 ml for the bleach test and 50 ml for the excess caustic and excess sodium carbonate test.

Grams per liter Available Chlorine
Reagents Fisher Catalog Number
Glacial acetic acid (50% by weight) - 1:1 (1 L) LC10290-2
Starch solution indicator (1 L) SS408-1
Sodium thiosulfate - 0.1N (1 L) SS368-1
KI crystals (500 g) P410-500
**Procedure**

- Add 50 ml of distilled water to an Erlenmeyer flask.
- Pipette a 5 ml aliquot of the bleach sample into the Erlenmeyer flask with stirring. Suggested aliquot of stock solution (25/250):

  - 3-10% sodium hypochlorite: 10 ml ... using 0.1 N Na₂S₂O₃ ... ~15 ml titrant
  - 10-16% sodium hypochlorite: 5 ml ... using 0.1 N Na₂S₂O₃ ... ~20-25 ml titrant
- Add 2 or 3 grams (1/2 teaspoon) of KI crystals and 10 ml of 1:1 acetic acid (in this order) to the Erlenmeyer flask.
- Titrate the solution with standardized sodium thiosulfate (0.1N) (Na₂S₂O₃) until the mixture is straw yellow in color. NOTE: A white sheet of paper should be placed under the Erlenmeyer flask to help see the color change. In addition a small high intensity light placed in the area of the Erlenmeyer flask pointing to the flask and on to the white paper should be used in order to best see the color change and to achieve accurate and repeatable results. The sodium thiosulfate must be added by drops and not a steady stream. If this slow addition of sodium thiosulfate is not done, the final results will not be accurate or repeatable.
- Add 5 ml of starch indicator and continue to very slowly, drop by drop, titrate the mixture until the blue color disappears.

**Calculation of Grams per Liter Available Chlorine**

Based on the following titration information:

Original Dilution: 25/250 NOTE: 25 ml of original bleach to 250 ml of aliquot solution
Sample Size: Assume 5 ml for example
Titrant (Sodium Thiosulfate): Variable NOTE: in actual calculations use the molarity number provided by the supplier. Assume titrant = 0.1009 M = 100.9 mM (for example)
Titrant Consumed (Na₂S₂O₃): Variable NOTE: For example assume 21.9 ml used during titration
Stoichiometry: 1/2 = 0.5
Molecular weight of Chlorine 70.91 grams/mole

The addition of acid to the titration flask changes the original chemical form of bleach from NaOCl to HOCl to Cl₂. Under these conditions, sodium thiosulfate can be used to reduce the valence of the chlorine atom from +1 to -1 (to the chloride ion - Cl⁻), a change of 2 electrons. This two electron change is accounted for in the calculation by the stoichiometry of 1 to 2 or 0.5.

\[
gpl \text{ of available chlorine} = \frac{(\text{ml of titrant}) \times (\text{mM of titrant}) \times \text{stoichiometry} \times (1 \text{ mole}) \times (70.91 \text{ grams})}{(\text{sample size} \times \text{original dilution}) \times (1000 \text{ mM}) \times (\text{mole})}
\]

Calculation examples from example numbers above

\[
21.9 \times 100.9 \times (0.5 \times 0.001 \times 70.91) = 156.71 \text{ gpl of available chlorine}
5^* \times (25/250)
\]

\[
21.9 \times 100.9 \times 0.03546 = 156.71 \text{ gpl of available chlorine}
5^* \times (25/250)
\]

*If 10 ml aliquot of the bleach sample is used to test strong bleach, change to sample size to 10 ml.

Conversion of grams per liter available chlorine to weight percent sodium hypochlorite or weight percent available chlorine requires an accurate measurement of the specific gravity using sample weight divided by 25 ml. Therefore, only grams per liter available chlorine should be used for specifications, comparison testing, and to price the value of the product.

When grams per liter available chlorine is used, there is a direct proportional relationship between the raw materials used. For example, the raw materials used to produce 120 gpl available chlorine versus 160 gpl available chlorine is a ratio of 1.333. Therefore, the value of the product has direct proportional relationship to the gpl available chlorine with the exception of delivery cost.
If the weight percent of sodium hypochlorite or available chlorine is used for final results, much greater errors in measurement will exist and the value of the product will not be directly proportional to the weight percent.

- **Weight percent of sodium hypochlorite (NaOCl) =** \( \frac{{gpl \ text{ available chlorine} \times 1.05}}{10 \times \text{specific gravity of sample}} \)

- **Weight percent of available chlorine =** \( \frac{\text{weight percent sodium hypochlorite}}{1.05} \)

- **Trade percent available chlorine =** \( \frac{\text{grams per liter available chlorine}}{10} \)

**Weight percent Sodium Hydroxide and Sodium Carbonate**

A variety of methods are currently being used by bleach manufacturers to measure excess caustic and alkalinity. These methods might include the use of barium to precipitate carbonate species or the use of various pH indicators during the titration steps. Powell Fabrication & Manufacturing, LLC recommends that the analytical methods below and it does not include a barium precipitation step. Phenolphthalein and methyl orange are used as pH indicators.

Barium precipitation is used by some laboratories in the weight percent sodium hydroxide and sodium carbonate titrations. Our experience has been that some type of chemical problem exists with the method and does not produce accurate results.

**Reagents**

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<td>Phenolphthalein indicator 1% (500 ml)</td>
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<td>Neutral hydrogen peroxide solution 30% (500 ml)</td>
<td>H325-500</td>
</tr>
<tr>
<td>Methyl orange indicator 0.1% (500 ml)</td>
<td>SM54-500</td>
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**Procedure**

- Add 50 ml of distilled water to an Erlenmeyer flask.
- Pipette a 50 ml of the aliquot bleach sample into the Erlenmeyer flask with stirring. This aliquot 50 ml bleach sample is from the same 25/250 dilution used in the available chlorine test.
- Slowly add 3 ml of neutral 30% \( \text{H}_2\text{O}_2 \) solution to the Erlenmeyer flask. The addition of 30% of \( \text{H}_2\text{O}_2 \) solution must be done carefully. The hydrogen peroxide is added to removed all of the sodium hypochlorite from the solution by converting it to salt and oxygen. Be aware that a higher bleach concentration will give a more vigorous reaction. This means the neutralization of 15% requires more care than adding \( \text{H}_2\text{O}_2 \) to a 5% bleach solution. Take the necessary precautions if more concentrated solution is used. Remember to stir the solution during the \( \text{H}_2\text{O}_2 \) addition failure to stir could lead to a sudden, vigorous reaction. To determine if enough hydrogen peroxide has been added, after the solution is carefully stirred and has then stopped to settle for 30 seconds, add one more drop of hydrogen peroxide to the solution. If no bubbles of oxygen are noted, no further addition of hydrogen peroxide is required. If oxygen bubbles are noted, continue to add drops of hydrogen peroxide until no oxygen is generated from the solution. Do not use 3% \( \text{H}_2\text{O}_2 \) since it may have free acid in the solution and the free acid will neutralize the excess caustic. Many times this free acid will greatly reduce the measured strength of excess caustic. Only use a acid free 30% \( \text{H}_2\text{O}_2 \).
- Add 5 drops of the phenolphthalein indicator and stir.
- Continue to stir and titrate the solution with standardized 0.1 N HCl until the pink color disappears. A white sheet of paper should be placed under the Erlenmeyer flask to help see the color change. In addition a small high intensity light placed in the area of the Erlenmeyer flask pointing to the flask and on to the white paper should be used in order to best see the color change and to achieve accurate and repeatable results. The 0.1 HCl must be added by drops and not a steady stream. If this slow addition of 0.1 HCl is not done, the final results will not be accurate or repeatable. Record the ml of acid used. \( N = \text{normality of standardized acid; A = ml acid used to titrate to the phenolphthalein endpoint.} \)
- Add 10 drops of the methyl orange indicator and stir.
- Continue to stir and titrate the solution very slowly, drop by drop, with standardized 0.1 N HCl until the yellow color changes to the first appearance of a “rust/red” color. This endpoint is not instantaneous. Stirring the titration, notice the “rust” color that appears as you drop HCl into the solution before it turns back to yellow. The first stable appearance of this rust color is what you are titrating to. Record the ml of acid used. \( B = \text{total ml acid used to titrate to the methyl orange endpoint.} \)
Calculations

Percent NaOH by weight: \( \frac{\left[ B - \left( B - A \right) \right] \times N \times 0.040 \times 100}{50/250 \times \text{wt of original sample}} \)

The addition of acid to the titration flask directly titrates the NaOH with HCl. Thus, the value 0.040 is the formula weight of NaOH (40) divided 1,000 because we are working in milliliters. The indicator, phenolphthalein, in base is red. After the base is titrated, the additional acid starts to change the mixture pH. When the pH changes from pH 10 to 8.2, the indicator changes from red to colorless indicating that all the base is titrated.

Percent Na₂CO₃ by weight: \( \frac{\left[ 2(B - A) \right] \times N \times 0.053 \times 100}{50/250 \times \text{wt of original sample}} \)

After the caustic titration is complete, the addition of more acid will titrate the carbonate ion (CO₃²⁻) to carbon dioxide (CO₂) a 2 electron change. Thus, the value 0.053 is the formula weight of Na₂CO₃ (106) divided by the electron change divide by 1,000 because we are working in milliliters. After the carbonate ion is titrated, the additional acid starts to change the mixture pH. When the pH changes for pH 4.4 to 3.1, the indicator changes from yellow to red indicating that all the carbonate ion is titrated.

Calculation of Liquid Bleach pH

The pH of liquid bleach can be calculated using the data from the weight percent sodium hypochlorite titration.

- Measure the liquid bleach specific gravity.
- Calculate the trade percent NaOH: 
  \[ \text{Trade percent NaOH} = \text{weight percent NaOH} \times \text{specific gravity} \]
- Calculate the g/L NaOH: 
  \[ g/L \text{NaOH} = \text{trade percent} \times 10 \]
- Calculate the moles/liter (M) of NaOH: 
  \[ M \text{NaOH} = \frac{g/L \text{NaOH}}{40} \]
- Calculate the pH: 
  \[ \text{pH} = 14 + \log (M \text{NaOH}) \]
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Weight Percent Sodium Chlorate

In basic solution, $\text{OCl}^-_1$ decomposition to form chlorate ion has been shown to be a second order process\(^1\) \(\text{Rate} = k_2 [\text{OCl}^-_1]^2\) with the following stoichiometry: \(3\text{OCl}^-_1 \rightarrow \text{ClO}_3^- + 2 \text{Cl}^-\).

The decomposition of $\text{OCl}^-_1$ involves chlorite ion ($\text{ClO}_2^-_1$) as an intermediate in the following generally accepted mechanism\(^2\).

\[
\begin{align*}
\text{OCl}^-_1 + \text{OCl}^-_1 & \rightarrow \text{ClO}_2^-_1 + \text{Cl}^- \\
\text{OCl}^-_1 + \text{OIC}_2^- & \rightarrow \text{ClO}_3^- + \text{Cl}^{-}
\end{align*}
\]


The most reliable $\text{ClO}_3^-$ measurement is by ion chromatography (IC). The titration method for $\text{ClO}_3^-$ suffers from many interferences and thus is not very accurate. We have an arrangement with an analytical laboratory to measure the various components in liquid bleach. Contact Dr. Bernard Bubnis at the address below.

Novachem Laboratories Inc.
5172 College Corner Pike
P.O. Box 638
Oxford, OH 45056
Ph: 513.523.3605 Fax: 513.523.4025

Bleach Strength Inorganics Transition Metal Ions
Weight percent Bleach Chlorate Ion Iron
Weight percent Caustic Bromate Ion Copper
Weight percent Alkalinity Nickel
Clarity/Suspended Solids

References

*Sodium Hypochlorite Safety and Handling Pamphlet*, The Chlorine Institute, Inc., Washington DC.
*Soda Bleach Solutions*, Diamond Shamrock, Cleveland, OH.
*Practical Guide to Chlorine Bleach Making*, Allied Chemical, Morristown, NJ.

Calculations

Grams per Liter of available Chlorine

Normality of $\text{Na}_2\text{S}_2\text{O}_3$ in mM = \(\text{__________ M x 1,000 = __________ mM}\)

ml $\text{Na}_2\text{S}_2\text{O}_3$ (titrant) consumed = \(\text{__________ ml}\)

Sample size of aliquot solution = \(\text{__________ ml}\)

Original Dilution = 25/250 strong bleach diluted to mark

\[
\frac{(\text{ml of titrant}) \times (\text{mM of titrant}) \times \text{stoichiometry} \times (1 \text{ mole}) \times (70.91 \text{ grams})}{(\text{Sample size} \times \text{original dilution}) \times (1,000 \text{mM}) \times \text{(mole)}}
\]

Typical calculation example form example numbers method above.

Normality of $\text{Na}_2\text{S}_2\text{O}_3$ in mM = 0.1009 M x 1,000 = 100.9 mM

ml $\text{Na}_2\text{S}_2\text{O}_3$ (titrant) consumed = 21.9 ml
Sample size of aliquot solution = 5 ml

Original dilution = 25/250 strong bleach diluted to mark

\[
\frac{21.9 \times 100.9}{5^* \times (25/250)} \times (0.5 \times 0.001 \times 70.91) = 156.71 \text{ gpl of available chlorine}
\]

\[
\frac{21.9 \times 100.9}{5^* \times (25/250)} \times 0.03546 = 156.71 \text{ gpl of available chlorine}
\]

*If 10 ml aliquot of the bleach sample is used to test strong bleach, change to sample size 10 ml.

**Percent NaOH by Weight**

Weight of original sample (25 ml) = \(\quad\) g (to nearest 0.001 g)

\(N = \) normality of standardized acid = \(\quad\) N

\(A = \) ml acid used to titrate to the phenolphthalein endpoint = \(\quad\) ml

\(B = \) ml acid used to titrate to the methyl orange endpoint = \(\quad\) ml

\[
\left\{\frac{\left(B - 2(B-A)\right)}{N} \times 0.040\right\} \times 100 \quad \text{50/250 x wt of original sample}
\]

\[
\frac{\left(\quad\right) - 2(\quad - \quad)}{0.1 \times 0.040 \times 100} = \quad\text{Weight percent NaOH}
\]

**Percent \(\text{Na}_2\text{CO}_3\) by Weight**

\[
\frac{\left[2(B-A)\right] \times N \times 0.053}{50/250 \times \text{wt of original sample}} \times 100
\]

\[
\frac{\left[2(\quad - \quad)\right] \times 0.1 \times 0.053 \times 100}{50/250 \times (\quad)} = \quad\text{Weight percent Na}_2\text{CO}_3
\]

**Specific Gravity of Solution**

Original 25 ml sample weight (grams) = \(\quad\) = \(\quad\) = \(\quad\)

\[
\frac{\text{Original 25 ml sample weight (grams)}}{25 \text{ ml}} = \frac{25}{25} = \quad\]