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## Foreword

This handbook outlines the methods for handling, storing, preparing and using soda bleach solutions. It includes information on the manufacture, physical properties and analytical methods for testing soda bleach solutions.

Additional information and contacts can be found at [www.oxychem.com](http://www.oxychem.com)

Occidental Chemical Corp.  
Basic Chemicals Group  
Occidental Tower  
5005 LBJ Freeway  
Dallas, TX 75244

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## Introduction

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This handbook provides information concerning proper procedures for the manufacture of sodium hypochlorite, or soda bleach, solutions. An attempt has been made to give a relatively comprehensive coverage of the subject. If additional technical information or specific recommendations regarding soda bleach solutions are desired, the Technical Service Group of Occidental Chemical Corporation will be pleased to render assistance. Requests for such information should be made to your local OxyChem representative.

Some safety and handling information has been taken directly from the Chlorine Institute's Pamphlet 96 with the permission of the Chlorine Institute. Pamphlet 96 also contains additional information on sodium hypochlorite.

For further information regarding caustic soda and chlorine, refer to the appropriate OxyChem handbook.

Sodium hypochlorite solutions have attained widespread usage in bleaching operations and as disinfectants, both in the home and in industry. A brief historical sketch may therefore be of interest.

Scheele, a Swedish chemist, is generally credited with discovering chlorine in 1774. During his experiments, he found that a solution of chlorine in water possessed definite bleaching properties. Since the reaction between chlorine and water forms hydrochloric and hypochlorous acids, early textile bleaching experiments were not successful because of damaged cloth.

In 1789, the French chemist Berthollet succeeded in chlorinating a solution of potash, forming a potassium hypochlorite solution. This solution proved to be a more successful bleach for textiles due to the absence of free hydrochloric acid. However, it never gained more than limited usage in the bleaching field, primarily because of the high cost of potash.

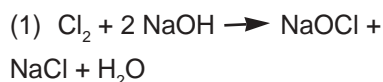
In 1798, Tennant of England prepared a solution of calcium hypochlorite by chlorinating a slurry of relatively inexpensive lime. The following year he patented a process for the manufacture of bleaching powder where chlorine gas was absorbed in a dry lime hydrate.

Labarraque succeeded, in 1820, in preparing sodium hypochlorite by chlorinating a solution of caustic soda. Varying concentrations of this solution have found a multitude of applications so that the general public is now well acquainted with the material. This handbook will discuss sodium hypochlorite solutions.

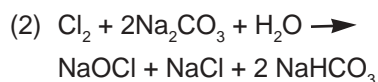
The term hypochlorites refers to the salts of hypochlorous acid (HOCl). Since the acid is extremely unstable, most users handle the more stable hypochlorite solutions instead. These salts are prepared by reacting chlorine with an alkali or an alkaline earth hydroxide.

## PREPARATION

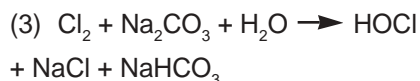
The common method for making sodium hypochlorite is to react chlorine with a solution of caustic soda. The final concentration of the sodium hypochlorite solution depends on the initial concentration of the starting caustic soda solution. The following equation gives the chemical reaction involved, regardless of concentration:



A more active, but less stable, sodium hypochlorite can be produced by chlorinating a solution of soda ash according to the following equation:



On further chlorination, hypochlorous acid will be produced:



Most commercial production processes react chlorine with caustic soda as in equation (1). This handbook addresses that method.

## STABILITY

Although more stable than hypochlorous acid, hypochlorites are inherently unstable themselves. They start breaking down when prepared and continue until completely decomposed. With proper care, the rate of decomposition can be controlled to the extent that relatively stable solutions can be prepared.

The stability and shelf life of a hypochlorite solution depends on five major factors:

- Hypochlorite concentration.
- Alkalinity or pH of the solution.
- Temperature of the solution, both in preparation and in storage.
- Concentration of certain impurities which catalyze decomposition.
- Exposure to light.
- Ionic strength of the solution.
- Contact with organic impurities.

Solutions containing low concentrations of hypochlorites decompose more slowly than those at higher concentrations. When stored under adverse conditions, solutions made at high concentrations soon may have a lower concentration than those which were originally made at low concentrations.

The alkalinity or pH has a pronounced effect on the stability of hypochlorite solutions. A pH value between 11.0 and 13.0 gives the most stable solutions. A slight excess of caustic soda also helps protect hypochlorite solutions from the harmful effects of light. To improve control, excess alkalinity should be adjusted based on the strength of bleach solution. Typical targets range from 0.2% excess alkalinity in a 20 grams per liter (gpl) bleach solution to 1.0% excess alkalinity in a 200 gpl bleach solution. There is no evidence that greater concentrations of excess alkalinity have a beneficial effect on the stability of hypochlorite solutions. In fact, excessively high alkalinity may damage textiles and retard the bleaching action of the hypochlorite.

## General Properties of Hypochlorites

Temperature during manufacture and storage definitely influences the stability of hypochlorite solutions. Care should be taken to keep solutions away from heat, as higher temperatures will increase the decomposition rate. Although low storage temperatures improve the stability of hypochlorite solutions, freezing should be avoided. Sodium hypochlorite solutions will freeze at different temperatures depending on the concentration of the solution.

The quality and stability of sodium hypochlorite solutions can be affected by the concentration of certain impurities. Trace metals such as nickel, copper and cobalt form insoluble metal oxides, which cause the bleach to catalytically decompose, forming oxygen gas and lowering bleach strength. These trace metals, as well as iron, calcium and magnesium, form sediment and may discolor the bleach solution.

Potential sources for these impurities include raw materials, processing equipment and product storage containers. The most common source for these metals, particularly nickel and copper, is the caustic soda feed. Diaphragm cell caustic soda typically contains a higher concentration of these metal catalysts than do membrane or rayon grades. However, many bleach manufacturers successfully make stable bleach

from diaphragm caustic soda.

Some techniques used by bleach producers to minimize the concentration of impurities in the finished product are listed below.

- Polish the finished bleach with a 0.5 to 1 micron filter. This will remove impurities which promote bleach decomposition and/or degrade the visual appearance.
- Use plastic or lined tanks and piping systems to reduce metal pick-up.
- Use soft water for the final dilution step.
- Allow finished bleach to settle until clear and decant before packaging.

The most effective of these, filtration of finished bleach, represents a method for removing insoluble oxides and other particulate matter from the finished bleach.

The type of filtration system required will depend on particulates loading, production rate and other considerations.

Light accelerates the decomposition of hypochlorite solutions. Avoid direct contact with sunlight. Opaque (non-translucent) containers for packaging hypochlorite solutions will reduce decomposition caused by light.

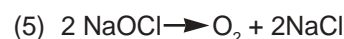
### DECOMPOSITION REACTIONS

Hypochlorite solutions decompose in two main ways: Effect of temperature and time:



In this reaction the sodium hypochlorite forms sodium chlorate and sodium chloride. The rate increases with increasing temperature. This reaction is not catalytic.

Effect of metal catalysts, temperature and light:



Trace metals such as nickel, copper and cobalt form insoluble metal oxides, which cause bleach to catalytically decompose to oxygen and sodium chloride. Light also catalyzes this reaction.

As discussed previously, pH has a pronounced effect on the stability of hypochlorite solutions. The breakdown of hypochlorite accelerates according to Reaction 4 as the pH value decreases. In addition, when the bleach solution becomes neutral, hypochlorous acid can form and release chlorine ( $\text{Cl}_2$ ).

# Terminology

The strength of soda bleach solutions is usually expressed as either available chlorine or sodium hypochlorite content. The term available chlorine is the amount of chlorine equivalent in oxidizing power to the hypochlorite present. Available chlorine is usually expressed as either percent by weight or grams per liter (gpl).

When describing concentration in terms of sodium hypochlorite content, the standard unit is per cent by weight.

It is important to specify the concentration units whenever describing the strength of bleach solutions. For example, 5.25 wt. % sodium hypochlorite is equivalent to 5.0 wt. % available chlorine, or 5.37 trade percent

### Conversions

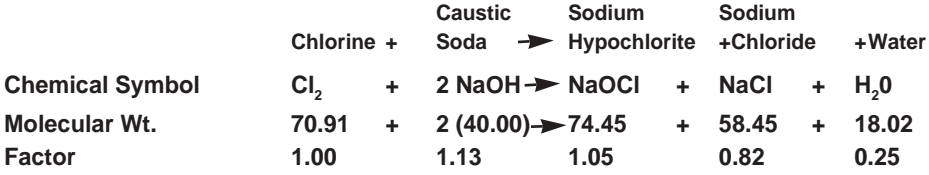
<b>Weight % available chlorine</b>	=	$\frac{\text{gpl available chlorine}}{10 \times \text{specific gravity of solution}}$
<b>Weight % sodium hypochlorite</b>	=	1.05 x wt. % available chlorine
<b>Weight % sodium hypochlorite</b>	=	$\frac{1.05 \times \text{gpl available chlorine}}{10 \times \text{specific gravity of solution}}$
<b>Trade Percent</b>	=	$\frac{\text{gpl available chlorine}}{10}$

# Manufacturing

Sodium hypochlorite (soda bleach) solutions can be prepared by reacting chlorine with solutions of caustic soda, soda ash (sodium carbonate), or a combination of caustic soda and soda ash. Soda ash processes produce less stable sodium hypochlorite solutions. For that reason, only the caustic soda processes will be discussed in this handbook.

Potassium hypochlorite, another bleach product, can be produced with the same equipment for production of soda bleach solutions.

As previously stated, chlorine will react with a caustic soda solution to produce sodium hypochlorite according to the following equation:



From the molecular weights of chemically pure materials, one pound of chlorine plus 1.13 pounds of caustic soda will produce 1.05 pounds of sodium hypochlorite. These calculations do not include any excess alkalinity needed in the resultant hypochlorite solution for stability.

Since commercial materials are not chemically pure, the foregoing calculations must be revised to take that into account. For practical pur-

poses, chlorine can be assumed to be 100% pure. Dry caustic soda is about 98% pure. The value of 1.13 divided by 0.98 gives a factor of 1.15 pounds of commercial caustic soda to react with one pound of chlorine. Since the strength of caustic soda solutions is given as actual concentration of sodium hydroxide content, the theoretical factor 1.13 will apply in calculations with caustic soda solutions.

## Manufacturing

The degree of hardness in water for dilution will influence the ratio of chlorine to caustic soda. Some caustic soda will be consumed by reacting with the hardness salts of calcium and magnesium in the water.

The exact ratio of chlorine and caustic soda will depend on the available water quality and the amount of excess caustic soda desired by the bleach manufacturer. The approximate amount of raw materials needed to produce given concentration of bleach solution can be calculated as follows:

### Chlorine

- (6) Pounds of  $\text{Cl}_2$  required = (wt. % available chlorine x specific gravity of bleach x 8.337 x gallons of bleach)  $\div$  100.  
 (7) Pounds of  $\text{Cl}_2$  required = gpl available chlorine x 0.008345 x gallons of bleach.

### Caustic Soda - Dry

- (8) Pounds of dry caustic soda required = (pounds of chlorine x 1.15) + (wt. % excess caustic soda x specific gravity of bleach x 8.337 x gallons of bleach)  $\div$  100.

### Caustic Soda - Liquid

- (9) Pounds of caustic soda solution required = [(pounds of  $\text{Cl}_2$  x 1.13) + (wt. % excess caustic soda x specific gravity of bleach x 8.337 x gallons of bleach)] / wt. % caustic soda solution.  
 (10) Gallons of liquid caustic soda required = pounds of liquid caustic soda  $\div$  (8.337 x specific gravity of caustic soda solution).

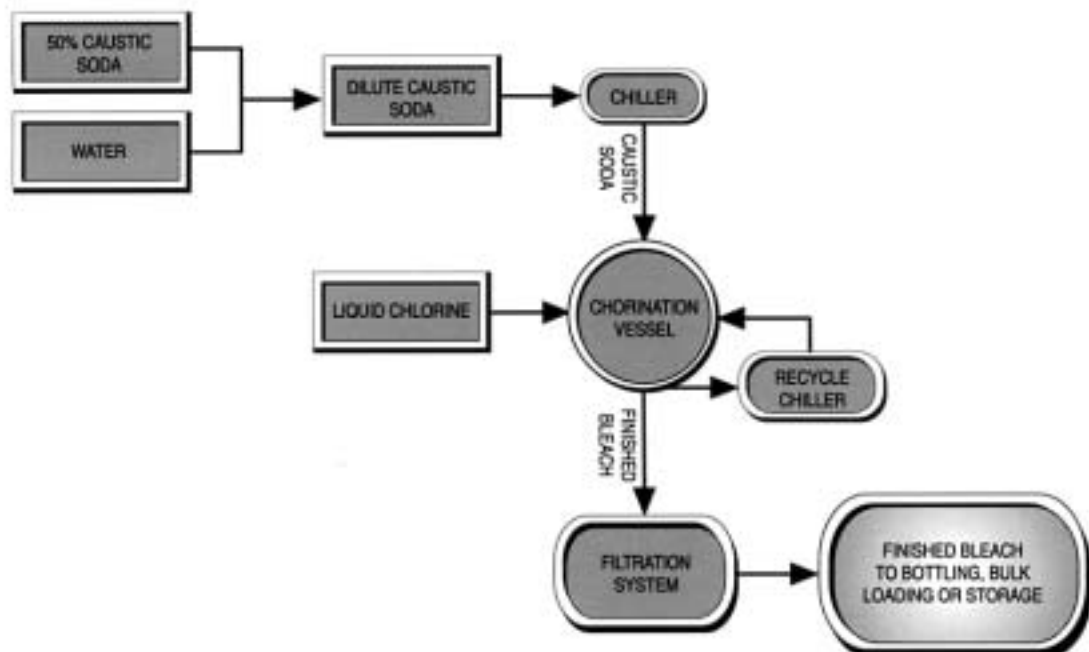
### Water

- (11) Pounds of water required = (gallons of bleach x 8.337 x specific gravity of bleach) - (pounds of  $\text{Cl}_2$  + actual pounds of caustic soda).  
 (12) Gallons of water required = pounds of water  $\div$  8.337.

*NOTE: All calculations are based on standard conditions at 60° F.*

## Typical Installation

**Figure 1**  
**Sodium Hypochlorite Manufacturing Process**



Tables 1 and 2 list the materials required for making 1000 gallons of bleach solution in concentrations from 10 to 200 gpl available chlorine. Table 1 should be used by those who express bleach concentration as gpl available chlorine. Table 2 will better serve those who express bleach concentrations as per cent by weight available chlorine or sodium hypochlorite. Tables 1 and 2 use rounded values and will vary slightly from calculated values.

## Example:

It is desired to make 1000 gallons of household bleach containing 5.25% sodium hypochlorite by weight. Table 2 provides the following information:

- (a) 5.25% sodium hypochlorite = 5.0% available chlorine by weight.
- (b) specific gravity = 1.075
- (c) desired excess caustic soda = 0.27%

(Reference equations on page 6).

$$(6) 5.0 \times 1.075 \times 8.337 \times 1000 \div 100 = 448 \text{ pounds of chlorine required.}$$

$$(8) (448 \times 1.15) + (0.27 \times 1.075 \times 8.337 \times 1000 \div 100) = 539 \text{ pounds of dry caustic soda required.}$$

$$(11) (1000 \times 8.337 \times 1.075) - (448 + 539) = 7975 \text{ pounds of water required.}$$

$$(12) 7975 \div 8.337 = 957 \text{ gallons of water required.}$$

Starting with 20% caustic soda (specific gravity = 1.223), instead of dry caustic soda, the calculation is:

$$(9) [(448 \times 1.13) + (0.27 \times 1.075 \times 8.337 \times 1000 \div 100)] \div 0.20 = 2652 \text{ pounds of 20% caustic soda solution required.}$$

$$(10) 2652 \div (8.337 \times 1.223) = 260 \text{ gallons of 20% caustic soda solution required.}$$

$$(11) (1000 \times 8.337 \times 1.075) - (448 + 2652) = 5862 \text{ pounds of water required.}$$

$$(12) 5862 \div 8.337 = 703 \text{ gallons of water required.}$$



# Manufacturing

**Table 1**  
**Materials for Making**  
**1000 Gallons of Bleach Solution**

Finished Bleach						Chlorine Required	Caustic Soda Required for Chlorination						Water Addition Required for Diluting Caustic Soda		
Strength		NaOCl Weight %	Specific Gravity	Pounds per Gallon	Excess Caustic NaOH%		Stoichiometric NaOH Pounds	Excess NaOH Pounds	TOTAL Pounds	Dry NaOH @ 98% Pounds	50% Liquid NaOH Gallons	Dilute Caustic Soda Solution For Chlorination		Dry NaOH Water Gal.	50% Liquid NaOH Water Gal.
Available Chlorine	Weight %											Solution Gallons	NaOH%		
10	0.99	1.04	1.014	8.454	0.10	83.5	94.4	8.5	102.8	104.9	16.1	989.4	1.23	991.4	979.1
20	1.95	2.04	1.028	8.570	0.20	166.9	188.6	17.1	205.7	209.9	32.2	980.2	2.45	982.8	958.3
30	2.88	3.02	1.042	8.687	0.21	250.4	283.0	18.2	301.2	307.3	47.1	972.2	3.57	975.1	939.3
40	3.79	3.98	1.056	8.804	0.23	333.8	377.2	20.2	397.4	405.6	62.1	964.4	4.69	967.3	920.1
50	4.67	4.91	1.070	8.921	0.25	417.3	471.5	22.3	493.9	503.9	77.2	956.7	5.81	959.5	900.9
60	5.54	5.81	1.084	9.037	0.33	500.7	565.8	29.8	595.6	607.8	93.1	948.7	6.98	951.0	880.3
70	6.38	6.69	1.098	9.154	0.40	584.2	660.1	36.6	696.8	711.0	109.0	941.2	8.13	942.6	860.0
80	7.19	7.55	1.112	9.271	0.47	667.6	754.4	43.6	798.0	814.2	124.8	933.8	9.28	934.3	839.6
90	7.99	8.39	1.126	9.387	0.54	751.1	848.7	50.7	899.4	917.8	140.6	926.6	10.41	925.8	819.1
100	8.77	9.21	1.140	9.504	0.61	834.5	943.0	58.0	1001.0	1021.4	156.5	919.7	11.55	917.4	798.6
110	9.53	10.01	1.154	9.621	0.65	918.0	1037.3	62.5	1099.9	1122.3	172.0	913.3	12.64	909.3	778.8
120	10.27	10.79	1.168	9.738	0.69	1001.4	1131.6	67.2	1198.8	1223.2	187.5	907.1	13.72	901.2	758.9
130	11.00	11.55	1.182	9.854	0.72	1084.9	1225.9	71.0	1296.9	1323.4	202.8	901.1	14.79	893.1	739.3
140	11.71	12.29	1.196	9.971	0.75	1168.3	1320.2	74.8	1395.0	1423.4	218.1	895.4	15.85	885.1	719.6
150	12.40	13.02	1.210	10.088	0.78	1251.8	1414.5	78.7	1493.2	1523.7	233.5	889.9	16.90	877.1	699.9
160	13.07	13.73	1.224	10.204	0.81	1335.2	1508.8	82.7	1591.4	1623.9	248.9	884.4	17.94	869.1	680.3
170	13.73	14.42	1.238	10.321	0.85	1418.7	1603.1	87.7	1690.9	1725.4	264.4	879.2	18.99	860.9	660.3
180	14.38	15.10	1.252	10.438	0.90	1502.1	1697.4	93.9	1791.3	1827.9	280.1	874.4	20.05	852.6	640.1
190	15.01	15.76	1.266	10.555	0.95	1585.6	1791.7	100.3	1892.0	1930.6	295.9	869.8	21.09	844.2	619.8
200	15.63	16.41	1.280	10.671	1.00	1669.0	1886.0	106.7	1992.7	2033.4	311.6	864.2	22.14	835.9	599.5

Table volumes based on standard conditions @ 60° F

\* (1) Trade Percent =  $\frac{\text{Grams Per Liter}}{10}$



**Table 2**  
**Materials for Making**  
**1000 Gallons of Bleach Solution**

Finished Bleach						Chlorine Required	Caustic Soda Required for Chlorination						Water Addition Required for Diluting Caustic Soda			
Strength		NaOCl Weight %	Specific Gravity	Pounds per Gallon	Excess Caustic NaOH%		Cl <sub>2</sub> Pounds	Stoichiometric NaOH Pounds	Excess NaOH Pounds	TOTAL Pounds	Dry NaOH @ 98% Pounds	50% Liquid NaOH Gallons	Dilute Caustic Soda Solution for Chlorination		Dry NaOH Water Gal.	50% Liquid NaOH Water Gal.
Available Chlorine	Grams per Liter * (1)												Solution Gallons	NaOH%		
1.0	10.1	1.05	1.014	8.455	0.10	84.6	95.6	8.5	104.1	106.2	16.3	989.5	1.24	991.3	978.9	
2.0	20.6	2.10	1.029	8.577	0.20	171.5	193.8	17.2	210.9	215.3	33.0	980.4	2.51	982.4	957.3	
3.0	31.3	3.15	1.044	8.702	0.21	261.1	295.0	18.3	313.3	319.7	49.0	972.7	3.71	974.1	936.9	
4.0	42.3	4.20	1.059	8.831	0.25	353.2	399.1	22.1	421.2	429.8	65.9	965.4	4.97	965.4	915.4	
5.0	53.7	5.25	1.075	8.964	0.27	448.2	506.5	24.2	530.7	541.5	83.0	958.1	6.23	956.5	893.5	
5.25	56.6	5.51	1.079	8.998	0.29	472.4	533.8	26.1	559.9	571.3	87.6	947.5	6.57	954.1	887.6	
5.50	59.5	5.78	1.083	9.031	0.31	496.8	561.4	28.0	589.4	601.4	92.2	937.3	6.91	951.6	881.5	
5.75	62.5	6.04	1.088	9.066	0.35	521.3	589.1	31.7	620.8	633.5	97.1	927.5	7.26	949.0	875.1	
6.0	65.4	6.30	1.092	9.101	0.37	546.0	617.0	33.7	650.7	663.9	101.7	917.9	7.61	946.5	869.0	
7.0	77.5	7.35	1.109	9.242	0.45	646.9	731.0	41.6	772.6	788.4	120.8	911.7	8.99	936.3	844.4	
8.0	90.0	8.40	1.126	9.387	0.54	751.0	848.6	50.7	899.3	917.7	140.6	906.3	10.41	925.8	818.9	
9.0	102.9	9.45	1.144	9.538	0.63	858.4	970.0	60.1	1030.1	1051.1	161.1	901.2	11.87	915.0	792.6	
10.0	116.1	10.50	1.163	9.693	0.67	969.3	1095.3	64.9	1160.2	1183.9	181.4	896.3	13.30	904.3	766.5	
11.0	129.9	11.55	1.182	9.853	0.72	1083.8	1224.7	70.9	1295.6	1322.1	202.6	892.0	14.78	893.2	739.4	
12.0	144.1	12.60	1.202	10.019	0.76	1202.2	1358.5	76.1	1434.6	1463.9	224.3	887.9	16.27	881.9	711.6	
13.0	158.7	13.65	1.222	10.189	0.80	1324.7	1496.9	81.5	1578.4	1610.6	246.8	884.0	17.81	870.1	682.8	
14.0	173.9	14.70	1.244	10.367	0.87	1451.4	1640.1	90.2	1730.3	1765.6	270.6	880.5	19.41	857.6	652.4	
15.0	189.6	15.75	1.266	10.550	0.95	1582.6	1788.3	100.2	1888.6	1927.1	295.3	877.6	21.06	844.5	620.6	

Table volumes based on standard conditions @ 60° F

\*(1) Trade Percent =  $\frac{\text{Grams Per Liter}}{10}$

# Manufacturing

**Table 3**  
**Amount of Bleach**  
**Produced From**  
**Standard Chlorine Containers**

Occasionally it is desirable to base the size of a batch of bleach on the entire contents of one or more chlorine containers. The volume of bleach produced at various concentrations from a 100-pound cylinder, a 150-pound cylinder or a ton container, may be determined by consulting Table 3 or Table 4.

(1) Trade Percent = gpl/10

Bleach Concentration	Gallons of Bleach Solution Produced with		
	gpl Available Chlorine (1)	100 lbs. Chlorine	150 lbs. Chlorine
10	1198	1796	23952
20	599	899	11983
30	399	599	7987
40	299	449	5992
50	240	359	4793
60	200	300	3994
70	171	257	3423
80	150	225	2996
90	133	200	2663
100	120	180	2397
110	109	163	2179
120	100	150	1997
130	92	138	1843
140	86	128	1712
150	80	120	1598
160	75	112	1498
170	70	106	1410
180	66	100	1331
190	63	95	1261
200	60	90	1198

**Table 4**  
Amount of Bleach  
Produced From  
Standard Chlorine Containers

Bleach Concentration	Gallons of Bleach Solution Produced with		
	Wt. % Available Chlorine	100 lbs. Chlorine	150 lbs. Chlorine
1.0	1182	1773	23641
2.0	583	875	11662
3.0	383	574	7660
4.0	283	425	5663
5.0	223	335	4462
5.25	212	318	4234
5.50	201	302	4026
5.75	192	288	3836
6.0	183	275	3663
7.0	155	232	3092
8.0	133	200	2663
9.0	116	175	2330
10.0	103	155	2063
11.0	92	138	1845
12.0	83	125	1664
13.0	75	113	1510
14.0	69	103	1378
15.0	63	95	1264

# Handling and Storage

## CAUSTIC SODA

Since metals are detrimental to the stability of bleach, caustic soda for bleach making should be handled to minimize metals dissolving into the solution.

Where 50% caustic soda is added directly into a chlorination tank or a continuous bleach system, the storage vessel should be lined to prevent iron contamination in the bleach.

Where 50% caustic soda is diluted to 25% or less with unsoftened water, then settled or filtered before transfer, the storage or dilution tanks can be plain steel. Any iron dissolved by the caustic soda will precipitate with the calcium and magnesium (hard water salts) after dilution, cooling, and settling or filtration.

Avoid any copper, zinc or aluminum in a caustic soda system. All are readily attacked by caustic soda. In addition, copper is an extremely active catalyst that can accelerate the decomposition of bleach.

Nickel is quickly attacked by bleach and greatly accelerates its decomposition. However, because of the almost total resistance to caustic soda under 300° F, nickel is an excellent material of construction for 50% caustic soda service.

Caustic soda tanks should have two outlet points. The opening for transfer of solution to process should be sufficiently off the bottom to prevent entrainment of sediment. Another outlet should be at the lowest point of the bottom of the tank to allow easy cleaning. Dilution tanks will accumulate a sediment caused by the precipitation of hard water salts and other metals.

A caustic soda dilution tank should be emptied and rinsed out at least annually to prevent sediment from accumulating close to the process solution outlet. Metals and other impurities concentrate in the sediment. A small amount of sediment unintentionally transferred to the chlorination vessel can provide enough contamination to produce poor quality bleach.

Steel tanks in caustic soda service become passivated with a gray-black film forming on the metal. Thereafter, caustic soda at less than 130° F will dissolve very little iron from the tank. The protective film should be preserved whenever practical. Therefore, when sediment is rinsed from a tank, only the bottom should be washed. Caustic Soda solution should then be put into the tank as soon as the washing is finished. Otherwise a soft rust will form and contaminate any future storage.

## SODIUM HYPOCHLORITE

### GENERAL

Few materials of construction will withstand the highly reactive nature of sodium hypochlorite. Improper selection of those materials may result in damage to the handling system and contamination of the product. As a general rule, no metals (with the exception of titanium and tantalum under certain circumstances) should be allowed to come in contact with this chemical.

### STORAGE

Warning - sodium hypochlorite solutions must be stored in vented containers, or in containers equipped with adequate relief devices. If venting rate is exceeded by the decomposition rate, swelling or damage to the container may occur.

## BULK STORAGE TANKS

Tank and lining manufacturer's products and processes vary considerably, therefore, selecting an appropriate storage vessel should be given thorough evaluation. Consultation with tank and lining suppliers is recommended.

### Rubber Lined Steel

Tanks of this type are generally custom fabricated for a specific process. They may be any size of shape depending on the needs of the user, but are typically closed vertical or horizontal cylindrical vessels from 1,000 to 30,000 gallons capacity.

### Fiberglass

The success or failure of this type of tank when used in sodium hypochlorite service depends upon a large number of variables including resin type and additives, type of reinforcement, fabrication technique, storage temperature, environmental exposure and the characteristics of the solution. While many tanks of this type are currently in use, it is advisable to deal only with fabricators having experience with sodium hypochlorite and who are willing to warranty the vessel for the intended applications.

Make certain tank has adequate UV (ultra violet) stabilizer or a gel coat outer layer designed for the area of intended use.

If possible, locate tank in a shaded area.

## Polyethylene Tanks

Although some sodium hypochlorite users have had success with polyethylene tanks, some suppliers will not certify their tanks for this use.

## TRANSFER SYSTEMS

### Materials Selection

The following materials are compatible with sodium hypochlorite solutions or as linings for non-compatible materials. Some may not be suitable for use in processes that manufacture sodium hypochlorite. Other materials not listed here may also be suitable.

1. PVDF (fluorinated polyvinylidene)
2. PTFE (polytetrafluoroethylene)
3. Titanium (Warning: titanium must not be used in contact with dry chlorine).
4. Ethylene Propylene Rubber
5. Chlorobutylene Rubber
6. Polypropylene
7. PVC (polyvinyl chloride)
8. CPVC (chlorinated polyvinyl chloride)
9. Tantalum
10. VitonA with a minimum durometer of 70

### Piping

The two factors which determine the selection of piping materials for sodium hypochlorite solutions are structural strength and chemical resistance. Where piping systems may be subject to physical stress, lined steel pipe should be selected. Lining types include polypropylene, PVDF, PTFE, or similar thermoplastics. In lighter stress situations, fiberglass and reinforced PVC is suitable. As with the fiberglass tanks, care should be exercised in the

selection of the resin for fiberglass piping. Where piping will not be subject to impact, Schedule 80 PVC or CPVC is often used. Conventional support spacing standards should be observed when using this type of piping system. When metal fittings must be used, titanium is the preferred material. Mild steel, stainless steel, and virtually all common metals will corrode rapidly on contact with sodium hypochlorite solutions. Additionally, the resulting corrosion products will contribute to product degradation. Alloy 20 has been reported to corrode in contact with sodium hypochlorite solutions causing product decomposition. Hastalloy C is also not recommended.

### Valves

Structural strength of the valve must be considered with respect to its specific application. Valve selection will depend upon the type of piping system being used.

### Pumps

Due to the numerous individual components comprising a complete pump, special care should be used when specifying this device. The centrifugal pump is the most common style found in sodium hypochlorite solution transfer systems. Casing and impellers may be of materials previously mentioned as chemically resistant to sodium hypochlorite. Impeller shafts should be made of titanium or protected by another compatible material. Pumps constructed of titanium are available and while more costly than "plastic" pumps will typically provide longer service.

## SYSTEM DESIGN

### Continuous Reactors For Manufacturing Bleach

Caustic soda and chlorine can be fed continuously into a mixing reactor to form bleach. Instrumentation sensing the oxidation reduction potential (ORP) of the bleach solution automatically controls the chlorination. The reactor should disperse the chlorine sufficiently in the caustic solution to allow total reaction before discharge.

One type of continuous reactor is a vertical column where caustic soda solution enters the top and chlorine enters the bottom. The rate of production can be varied by adjusting the caustic soda feed rate or through a level sensing device in the storage tank. ORP instrumentation controlling chlorine flow is often linked with a warning system to signal the operator if the automatic valve should fail to control the chlorine properly.

Recycling part of the stream through the reactor helps to smooth out the process fluctuations.

### Discharge Systems

As previously noted, sodium hypochlorite solutions tend to deteriorate with age, leaving a sediment in the bottom of storage vessels. To avoid transferring this sediment into the process, the primary tank discharge nozzle should be located 1 to 3 feet from the bottom of the tank. An additional discharge nozzle should be located as close to the bottom as possible, (or on the bottom if the tank is elevated), to allow for completed drainage and periodic cleaning.

## Handling and Storage

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Where product quality is critical, filter systems are available to remove virtually all sediments. In some applications, the process can tolerate this sediment if it is continually transferred out of the storage tank along with the sodium hypochlorite solution. Cone bottom tanks can facilitate removal of the sediment. In other applications the process cannot tolerate this sediment and it cannot be allowed to accumulate.

### Venting/Overflow System

The worst case condition for the vent sizing is usually the venting rate required due to decomposition of the contents of the storage vessel. The vent sizing required for discharging or filling is a secondary To eliminate excessive pressure or vacuum build-up when filling or discharging the tank, a venting system must be provided. As a minimum, this system should contain a nozzle at the top of the tank. It should be sized to prevent excessive vacuum or pressure when the tank is discharging or filling. When filling the tank from bulk tank trucks under air pressure, large "air hammers" may occur. Therefore, vent piping should be rigidly secured to prevent vibration. The tank should also have a nozzle on the side near the top. This nozzle should be sized to release

the entire filling rate without reaching the tank's vent. Piping should be installed to direct the overflowing solution away from personnel into a containment area.

### Gauging Devices

Some tanks are sufficiently translucent to allow for visual gauging from level markers painted on or molded into the side of the tanks. Where lighting conditions or tank construction do not permit this method of gauging, external gauging systems must be provided. Differential pressure systems have been used successfully. Manometers and sight glass gauges are also used but require additional liquid filled connections, thus potential failure points on the tank. An independent, back up level sensor should be used to prevent tank overflow in the event of a level gauge failure.

# Safety in Handling Sodium Hypochlorite

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## Read the MSDS before use

Sodium hypochlorite solution is normally a light yellow liquid with a characteristic bleach odor. Sodium hypochlorite is unstable and can release chlorine gas if acidified. To improve the stability of hypochlorite solutions an excess alkalinity is usually maintained. Hypochlorite solutions are corrosive to eyes, skin and mucous membranes.

## PRECAUTIONS

- Emergency shower and eye-wash facility should be in close proximity to where sodium hypochlorite solution is handled.
- Insure adequate ventilation or use a NIOSH approved respirator with an acid gas cartridge with a dust, fume and mist filter where airborne concentrations are expected to exceed exposure limits or when symptoms have been observed that are indicative of overexposure.
- Avoid breathing fumes.
- Avoid contact with eyes, skin and clothing.
- Wash thoroughly after handling.
- Wear goggles and faceshield, plus chemical resistant gloves made of rubber, neoprene or vinyl. Wear chemical resistant clothing and boots if splashing or contact may occur.
- Do not allow contact with organic materials such as rags, wood fibers, paper, debris, or with reducing chemicals except under controlled conditions.
- Do not discard indiscriminately. A spontaneous combustion fire could result.

- Do not mix with acids, ammonia, heavy metals, ethers or reducing agents. To do so may release hazardous gases.
- Store in corrosion-resistant tanks.

## FIRST AID

### Eyes:

IMMEDIATELY FLUSH EYES WITH A DIRECTED STREAM OF WATER for at least 15 minutes, forcibly holding eyelids apart to ensure complete irrigation of all eye and lid tissue. Washing eyes within several seconds is essential to achieve maximum effectiveness.

### Skin:

Flush thoroughly with cool water under shower while removing contaminated clothing and shoes. Discard non-rubber shoes. Wash clothing before reuse.

### Inhalation:

Remove to fresh air. If breathing is difficult, have trained person administer oxygen. If respiration stops, have a trained person administer artificial respiration.

### Ingestion:

NEVER GIVE ANYTHING TO AN UNCONSCIOUS PERSON. If swallowed, DO NOT INDUCE VOMITING, although it may occur spontaneously. Give large quantities of water. If available, give several glasses of milk. Sodium bicarbonate, which would generate carbon dioxide should not be used. Keep airways clear.

**GET MEDICAL ATTENTION IMMEDIATELY.**

## FIRE

Use water or other extinguishing medium appropriate for surrounding fire. May release toxic fumes under fire conditions. Wear NIOSH/MSHA approved positive pressure self-contained breathing apparatus and full protective clothing.

## SPILL

NEVER FLUSH TO SEWER.

Contain spill with dike to prevent entry into sewers or waterways. For small spills, absorb with inorganic absorbents. Flush spill area with water ONLY IF water can be collected, and place in appropriate container for proper disposal. For large spills, dike and pump into properly labeled containers for proper disposal.

Report release, if required, to the appropriate local, state and federal agencies.

**Note:** For additional information refer to OxyChem Handbooks on Chlorine & Caustic Soda, in addition to the MSDS on Chlorine, Caustic Soda and Sodium Hypochlorite.



# Methods of Analysis

## DETERMINATION OF SODIUM HYPOCHLORITE, SODIUM HYDROXIDE AND SODIUM CARBONATE IN CAUSTIC SODA BLEACH SOLUTIONS

### PURPOSE

To provide a means for quantifying sodium hypochlorite, sodium hydroxide and sodium carbonate levels in caustic soda bleach solutions.

### THEORY

Sodium hypochlorite (NaOCl) is the active agent in caustic soda bleach solutions. Chemically, hypochlorites are the salts of hypochlorous acid (HOCl) and are inherently unstable. The stability of a hypochlorite solution is dependent on five major factors.

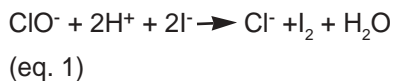
1. Hypochlorite concentration.
2. Alkalinity (pH) of the solution.
3. Temperature of the solution, both in production and storage.
4. Concentration of impurities which catalyze decomposition.
5. Exposure to light.

Any one of the above factors or combination of factors can affect the strength of a caustic soda bleach solution. Therefore, reliable methods of quantifying the sodium hypochlorite, sodium hydroxide and sodium carbonate concentrations of a caustic soda bleach solution are necessary.

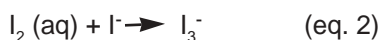
### A) Hypochlorite Determination

Hypochlorite concentration is

determined by iodometric titration. In this method, the hypochlorite ion is first reacted with excess I<sup>-</sup> in an acidic medium to form an equivalent amount of iodine:



Molecular iodine reacts with excess iodide in solution to form tri-iodide via the following reaction:

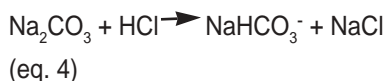


Each mole of tri-iodide is then titrated with two moles of sodium thiosulfate and the concentration of tri-iodide is determined. The percent hypochlorite in the sample can then be calculated from the tri-iodide concentration.

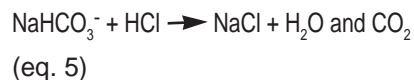


### B) Sodium Hydroxide and Sodium Carbonate Determination

Sodium hydroxide and sodium carbonate are quantified by a two-step titration with a standardized acid solution. During the first step, the hydroxide is neutralized and the carbonate is converted to bicarbonate simultaneously via the following reactions:



When both of these reactions (eqs 3 & 4) come to completion, an endpoint is indicated. The solution is then further titrated with acid to convert the bicarbonate to water and carbon dioxide.



A second endpoint is indicated at the completion of this reaction. The volumes of acid used in these two steps are then used to calculate the percent sodium hydroxide and sodium carbonate in the sample.

### APPARATUS

Analytical Balance; 200g + 0.0001 (Mettler At-200 or equivalent).

250 ml. Erlenmeyer Flask; (Fisher Scientific #10-090B or equiv.)

Magnetic Stirrer; (Fisher Scientific #14-493-120-S or equiv.)

Magnetic Stirring Bars; (Fisher Scientific #14-511-62 or equiv.)

250 ml. Volumetric Flask; Class A Volumetric, (Fisher Scientific #10-210E or equiv.)

5 ml Pipet; Class A Volumetric, (Fisher Scientific #13-650-2F or equiv.)

10 ml. Pipet; Class A Volumetric, (Fisher Scientific #13-650-2L or equiv.)

20 ml. Pipet; Class A Volumetric, (Fisher Scientific #13-650-2N or equiv.)

25 ml. Pipet; Class A Volumetric, (Fisher Scientific #13-650-2P or equiv.)

50 ml. Pipet; Class A Volumetric, (Fisher Scientific #13-650-2S or equiv.)

50 ml. Pipet; Class A Volumetric, (Fisher Scientific #03-700-2C or equiv.)

### REAGENTS

#### Deionized water

1:1 Acetic Acid; Add 500 ml of ACS reagent grade Glacial Acetic Acid (Fisher Cat# A38 or equiv.) to 500 ml of deionized water.

10% Potassium Iodide solution; Weigh 100.0 grams of KI (Fisher Cat#P412, or equiv.) into a one liter volumetric flask, add deionized water and mix to dissolve. Dilute with deionized water to volume.

0.1 N Sodium Thiosulfate solution;

Weigh 24.8190 grams of  $\text{Na}_2\text{S}_2\text{O}_3$  (Fisher Cat#S445, or equiv.) into a one liter volumetric flask, add deionized water and mix to dissolve. Dilute with deionized water to volume. Standardize the solution before use (see Standardization section). Standardized 0.1 N  $\text{Na}_2\text{S}_2\text{O}_3$  is also commercially available from Fisher Cat. #SS368-1. Starch Indicator; 1% solution - (Fisher Cat# SS48-1, or equiv.) Phenolphthalein Indicator solution, 1% - Fisher Cat# SP62-500 Methyl Orange Indicator, 0.1%; (Fisher Cat# SM54-500, or equiv.) 0.1 N Hydrochloric Acid solution; Transfer 8.3 ml of ACS reagent grade concentrated hydrochloric acid (Fisher Cat# A144, or equiv.) into a one liter volumetric containing deionized water, add additional water to bring to volume. Standardize the solution before use (see Standardization section). Standardized 0.1 N HCl is also commercially available from Fisher Cat. #SA54-1. 3% Hydrogen Peroxide solution; (Fisher Cat# H312-500, or equiv.)

## SAFETY

Refer to the MSDS for the proper handling procedures for all chemicals being analyzed by this method.

Caustic soda bleach solutions are irritating to the eyes and skin. Potassium iodide is toxic and sodium thiosulfate is an irritant, both should be handled with care. Acetic acid and hydrochloric acid are extremely corrosive. If any of these chemicals comes in contact with the eyes or skin, the affected area should be flushed with plenty of clean water for a minimum of 15 minutes. **Seek medical attention immediately.**

## PROCEDURE

### Sodium Hypochlorite Determination

1. Pipet 25 ml. of caustic soda bleach solution into a 250 ml. volumetric flask. Record the sample weight to the nearest 0.01 g. Add deionized water to the 250 ml. mark and mix thoroughly. This sample solution will be used as a stock solution for the determination of %NaOCl, % NaOH and %  $\text{Na}_2\text{CO}_3$ .  
**Note 1:** Sample sizes recommended in this method are for commercial strength bleach solutions (approximately 5.25% NaOCl). For samples of bleach with solution strengths different from commercial grade products, adjustments to sample size or aliquot size may be necessary.
2. Pipet a 10 ml aliquot of the stock solution into a 250 ml. Erlenmeyer flask containing 50 ml. of deionized water.
3. Add 25 ml. of 10% potassium iodide solution. The sample solution will change from clear to an intense yellow color.
4. Add 10 ml. of 1:1 acetic acid. Addition of acetic acid to a solution containing iodide liberates iodine which results in a further color change to amber brown.
5. Place the mixture on a magnetic stirring apparatus and gently stir.
6. Titrate using 0.1 N sodium thiosulfate to a straw yellow color, taking care to not over-titrate the sample to clear.
7. Add 5 ml. of starch indicator and continue the titration until the blue color disappears. Starch indicator reacts with iodine to form a very intense blue/purple color complex, which is visible at very low concentrations of iodine ( $2 \times 10^{-5}$  M).
8. Record the volume (ml). of 0.1 N sodium thiosulfate used. This volume will be used to calculate the % NaOCl in the sample.

### Sodium Hydroxide and Sodium Carbonate Determination

1. Pipet a 50 ml. aliquot of the sample stock solution (see Step 1 of sodium hypochlorite determination) into 250 ml. Erlenmeyer flask containing 50 ml. of deionized water.
2. Add 20 ml. of neutral 3% hydrogen peroxide solution and cool the sample to 0° to 5° C. The addition of peroxide is necessary to neutralize the sodium hypochlorite in the sample aliquot.
3. Add 3 drops of phenolphthalein indicator and titrate with 0.1 hydrochloric acid solution until the pink color disappears.
4. Record the volume of acid used to the nearest 0.02 ml. This volume will be used to determine the percent sodium hydroxide present in the sample solution.
5. Add 3 drops of methyl orange indicator to the same sample solution and continue to titrate with 0.1 N hydrochloric acid solution until the yellow color changes to red. Take care to titrate slowly since very little acid will be required to produce the second endpoint.
6. Record the volume of acid used to the nearest 0.02 ml. This volume will be used to determine the percent sodium carbonate present in the sample solution.

## CALCULATIONS

A) % Sodium Hypochlorite

Let:

W=Weight (g) of original sample.

V=Volume (ml) of 0.1 N sodium thiosulfate solution

N=Normality of the sodium thiosulfate solution

$10/250$ =Dilution factor from stock solution, i.e. - 10 ml aliquot taken from 250 ml stock

0.03723=milliequivalent weight of NaOCl

# Methods of Analysis

0.03545=milliequivalent weight of  $\text{Cl}_2$

The sodium hypochlorite content can be calculated as wt%

NaOCl or as wt% Available Chlorine

$$\% \text{NaOCl} = \frac{(V)(N)(0.03722)(100)}{10/250 \times W}$$

%Available Chlorine=

$$\frac{(v)(N)(0.03545)(100)}{10/250 \times W}$$

B) % Sodium Hydroxide and Sodium Carbonate Determination

Let:

$W_1$ =Weight (g) of original sample.

$V_1$ =Volume (ml) of 0.1 N HCl needed to reach the phenolphthalein endpoint

$V_2$ =Volume (ml) of 0.1 N HCl needed to reach the methyl orange endpoint

N=Normality of the hydrochloric

0.040= The milliequivalent weight of NaOH

0.053= The milliequivalent weight of  $\text{Na}_2\text{CO}_3$

50/250=Dilution factor from stock solution, i.e - 50 ml aliquot taken from 250ml stock

$V_1$  is the amount of acid required to neutralize the hydroxide and convert the carbonate to bicarbonate as shown in equations 3 and 4.

$V_2-V_1$  is the additional volume of acid required to convert the bicarbonate to carbon dioxide and water. The overall titration of sodium carbonate with hydrochloric acid requires two moles of HCl for each mole of  $\text{Na}_2\text{CO}_3$  since one mole is needed to convert  $\text{Na}_2\text{CO}_3$  to  $\text{NaHCO}_3$  and another mole is required to convert the  $\text{NaHCO}_3$  to  $\text{CO}_2$ .

therefore

$2 \times (V_2-V_1)$ = Volume of acid that reacted with  $\text{Na}_2\text{CO}_3$  in the sample

and

$V_2 - [2 \times V_2 - V_1]$ = Volume of acid that reacted with the NaOH in the sample

$$\% \text{NaOH} = \frac{(V_1) - [2(V_2 - V_1)](N)(0.040)(100)}{50/250 \times W}$$

$$\% \text{Na}_2\text{CO}_3 = \frac{2(V_2 - V_1)(N)(0.053)(100)}{50/250 \times W}$$

## QUALITY ASSURANCE

Clean all apparatus before use to eliminate contamination.

Duplicate analysis should be performed on a minimum of 10% of samples analyzed. Results should be reproducible within 0.025%.

Concentrations of sodium hypochlorite, sodium hydroxide, and sodium carbonate found in samples should be compared with the manufacturers specifications (if available) to ensure the product meets these standards.

Hydrochloric acid and sodium thio-sulfate solutions should be standardized at least monthly.

## REFERENCES

1. ASTM Volume 15.04, D 2022 (with Modifications)
2. Vogel, Arthur I., A Textbook of Qualitative Inorganic Analysis, 3rd edition, 1961

## STANDARDIZATION OF 0.1N HYDROCHLORIC ACID USING SODIUM CARBONATE AND MODIFIED

## METHYL ORANGE INDICATOR

### APPARATUS

Buret: 100ml, Class A, Fischer Cat# 03-700-22D or equiv.

Erlenmeyer Flasks; 250ml, Fischer Cat# 10-090B or equiv

Weighing Dish; disposable, Fischer Cat# 02-202A or equiv.

Stirring Bars; 38mm x 8 mm, Fischer Cat# 14-511-64 or equiv.

Analytical Balance;

Mettler AT 200 or equiv.

### REAGENTS

0.1N Hydrochloric Acid; measure 8.3mL of concentrated hydrochloric acid (sp.gr. 1.19) into a graduated cylinder and transfer it to a 1L volumetric flask. Dilute to the mark with water, mix well and store in a tightly closed container. A prepared solution of 0.1N HCl can also be purchased (Fischer Scientific Cat# SA54-1 or equiv.)

Sodium Carbonate; anhydrous, volumetric standard grade, EM Science Cat# 6394-2. Dry at 250° C in a platinum or porcelain crucible for 4 hrs.

Water, Deionized; this water should be carbon dioxide free - freshly boiled and cooled or purged with nitrogen for two hours.

Modified Methyl Orange Indicator; dissolve 0.14 gm of methyl orange and 0.12 gm of Xylene Cyanole FF in water and dilute to 100 ml.

**SAFETY**

Refer to MSDS for the proper handling procedures for each of the chemicals listed in this procedure.

Hydrochloric acid is a strong acid, it is corrosive to body tissue and can cause immediate and severe burns to eyes. Wear proper gloves, proper eye protection and other protective clothing when handling.

When handling sodium carbonate, avoid inhalation or contact with skin.

Xylene Cyanole is a flammable solid. Use proper ventilation, avoid prolonged breathing of vapors or prolonged or repeated contact with skin.

**STANDARDIZATION PROCEDURE**

Weigh 1.0 grams of sodium carbonate to the nearest 0.0001 grams into a weighing dish. Carefully transfer this material to an Erlenmeyer flask, add 75 ml of deionized water and swirl to dissolve. Add 3 drops of the modified orange indicator and titrate with the HCl solution to a magenta color change.

Repeat the above titration procedure on at least three more solutions of sodium carbonate.

**CALCULATIONS**

The following is the formula used to calculate the normality of the hydrochloric acid.

Let:

N = Normality of HCl

W = Weight (in grams) of Na<sub>2</sub>CO<sub>3</sub> used

V = Volume of HCl required

Milliequivalent wt. of Na<sub>2</sub>CO<sub>3</sub> = 0.053

$$N = \frac{W}{(V) \times (0.053)}$$

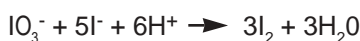
Calculate the average the normality of hydrochloric acid from the individual values. Also calculate the standard deviation and percent relative standard deviation (%RSD) for the standardization procedure.

**STABILITY**

Restandardize monthly or sooner.

**STANDARDIZATION OF 0.1N SODIUM THIOSULFATE WITH POTASSIUM IODATE****THEORY**

A solution of sodium thiosulfate can be standardized by titrating it into an acid solution containing a known amount of potassium iodate and a starch indicator. The acid reacts with the iodate to form iodine. The iodine is stoichiometrically reduced by the thiosulfate. The endpoint of the reaction is indicated when the solution changes from a blue color to colorless. 6 moles of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> are required to react with 1 mole of KIO<sub>3</sub>

**APPARATUS**

Analytical Balance; 200g±0.0001 (Mettler At-200 or equiv.)

250 ml. Erlenmeyer Flask; wide mouth, (Fisher Cat#: 10-090B or equiv.)

Magnetic Stirrer; (Fisher

Cat#: 14-493-120MR or equiv.)

Magnetic Stirring Bar; 1 1/2' x 5/16' dia, (Fisher Cat#: 14-511-64 or equiv.)

250 ml. Volumetric Flask; Class A Volumetric, Fisher Cat#: 10-210E or equiv.)

2 ml. Pipet; Class A Volumetric, (Fisher Cat:# 13-650-2C or equiv.)

5 ml Pipet; Class A Volumetric, (Fisher Cat: #: 13-650-2F or equiv.)

50 ml. Buret; Class A Volumetric, (Fisher Cat: #: 03-700-2C or equiv.)

**REAGENTS**

Deionized water

0.1 N Sodium Thiosulfate solution:

Weigh 25 grams of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> 5H<sub>2</sub>O (Fisher Cat#: S445 or equiv.) into a one liter volumetric flask, add deionized water and mix to dissolve.

Dilute with deionized water to volume. Store the solution in a tightly capped amber bottle.

Potassium Iodide; iodate free (Fisher Cat#: P410-500, or equiv.)

Potassium Iodate; dried at 120° C for at least one hour (Fisher Cat:#: P253-500, or equiv.)

2 N Sulfuric Acid solution; Weigh 55.6 of ACS reagent grade concentrated sulfuric acid (Fisher Cat#: A300-212, or equiv.) into a one liter volumetric flask containing 500 ml of deionized water, mix, allow to cool and diluted to volume with deionized water.

Starch Indicator - 1% solution; (Fisher Cat#: SS408-1, or equiv.)

Phenolphthalein Indicator - 1% solution;

(Fisher Cat#: SP62-500

Methyl Orange Indicator; (Fisher Cat#: SM54-500, or equiv.)

**SAFETY**

Refer to the MSDS for the proper handling procedures for all chemicals being analyzed by this method.



# Methods of Analysis

Potassium iodate is an oxidizer and should be handled accordingly. Potassium iodide is toxic and sodium thiosulfate is an irritant, both should be handled with care. Sulfuric acid is extremely corrosive. If any of these chemicals comes in contact with the eyes or skin, the affected area should be flushed with plenty of clean water for a minimum of 15 minutes. **Seek medical attention immediately.**

## PROCEDURE

1. Weigh 0.14 - 0.15 grams of dried potassium iodate to the nearest 0.0001 grams, transfer to a 250 Erlenmeyer flask and dissolve in 50 ml. of deionized water.
2. Add 2.0 grams of iodate free potassium iodide and 5 ml. of 2N sulfuric acid.

**Note:** To determine if the KI is iodate free, dissolve a small portion of the reagent in 2N sulfuric acid. No immediate yellow color should be observed. Add 1-2 ml of starch indicator, if no immediate blue color is produced, the potassium iodide is iodate free.

3. Place the mixture on a magnetic stirring apparatus and gently stir.
4. Titrate using 0.1 N sodium thiosulfate to a straw-yellow color, taking care to not over-titrate the sample to clear.
5. Add approximately 100 ml. of deionized water and 2.0 ml. of starch indicator and continue the titration until the blue color disappears. To achieve accurate results, the addition of the sodium thiosulfate titrant should be done very slowly. The endpoint of the titration is very sharp (color changes from

dark blue or purple to clear) and dropwise addition is recommended. Record the volume of sodium thiosulfate used to the nearest 0.02 ml.

6. Repeat the titration with two more accurately weighted portions of potassium iodate and record the volume of sodium thiosulfate used to the nearest 0.02 ml. for each portion.

## CALCULATIONS

The following formula is used to calculate the normality of the sodium thiosulfate.

Let:

N=Normality of the sodium thiosulfate solution.

W=Weight (g) of potassium iodate

V=Volume (ml) of 0.1 N sodium thiosulfate needed to reach the clear endpoint 0.03567 = milliequivalent weight of  $KIO_3$ .

$$N = \frac{W}{V \times 0.03567}$$

Average the values obtained from the three titration and also calculate the standard deviation and percent relative standard deviation (% RSD) of the standardization procedure.

## STABILITY

Sodium thiosulfate solutions are relatively stable, but do decompose over time. Exposure to air (especially carbon dioxide), light and airborne bacteria will accelerate the decomposition reaction. Therefore, restandardization should be performed on monthly basis or sooner.

## REFERENCES

1. ASTM 15.04 D 2022 (With Modifications)
2. Vogel, Arthur I., A Textbook of Qualitative Inorganic Analysis, 3rd edition, 1961.

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