**Chloramine Decay Test Protocol**
**Charlotte Smith & Associates, Inc.**
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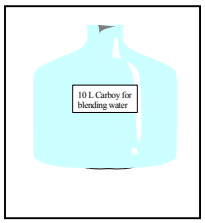
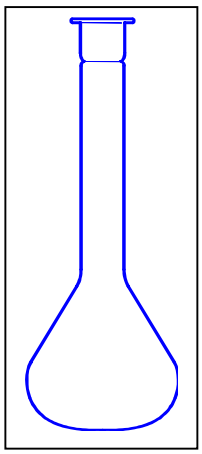
Pre-treat glassware and carboys with a 4 mg/L chlorine solution, rinse 5 times with distilled water, and dry in drying oven. See Table 1 for list of glassware.

1. Collect water in pre-treated 6 liter glass Erlenmeyer flasks or 5 gallon polyethylene carboys.




2. Blend the water according to the desired *blend ratio* (as shown on Table 2).

3. Prepare 1 liter of 1000 mg/L (1 mg/mL) sodium hypochlorite solution from Water Treatment Plant's stock hypochlorite. Obtain information regarding hypochlorite stock's percent active ingredient from plant staff, or use a hydrometer.

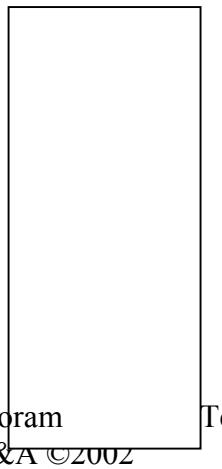


If: Plant's sodium hypochlorite solution is 12.3% active ingredient:

Then: (Density of water = 1 kg/L = 1000 mg/mL * specific gravity of the hypochlorite stock * % by weight of hypochlorite stock * molecular proportion of $Cl_2$/NaOCl = 70 / 23 + 16 + 35 = 0.95).
1,000 mg/mL * 1.25 * 0.123 * 0.95 = 146.1 mg/mL

$C_1 * V_1 = C_2 * V_2$
$C_1$ = Plant Stock's concentration = 146.1 mg/mL
$C_2$ = Final desired concentration of working stock 1 mg/mL
$V_1$ = Amount of Plant's stock to add to 1 liter DI $H_2O$ (to be calculated)
$V_2$ = 1000 mL

$V_1$ = 6.84 mL



4. Prepare 1 liter of 1000 mg/L (1mg/mL) ammonium hydroxide from reagent grade supply.

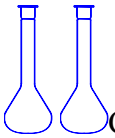
If: The reagent grade ammonium hydroxide is 29.2% active ingredient (as ammonia-N).
Then: Density of water * specific gravity of the ammonium hydroxide stock * % by weight of ammonium hydroxide stock * molecular proportion of N/$NH_3$-N = 14 / 17 = 0.82).

1,000 mg/mL * 0.898 * 0.292 * 0.82 = 221.7 mg/mL $NH_3$-N

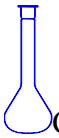
$C_1 * V_1 = C_2 * V_2$

$C_1$ = Plant Stock's concentration = 221.72 mg/mL
$C_2$ = Final desired concentration of working stock 1 mg/mL
$V_1$ = Amount of Plant's stock to add to 1 liter DI $H_2O$ (to be calculated)
$V_2$ = 1,000 mL

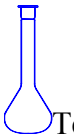
$V_1$ = 4.65 mL



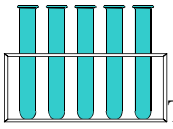
5. Check free and total chlorine concentrations of the sodium hypochlorite stock solution by diluting 1 ml and 2 mL into 2 separate 1000 mL volumetric flasks brought to 1,000 mL with DI $H_2O$. If stock solution is not 1 mg/mL remake stock solution or adjust future calculations based on actual stock solution concentration. See Table 1 for a list of reagents.



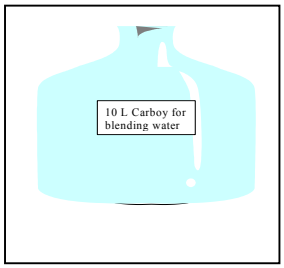
6. Check free ammonia concentration of ammonia-N stock solution by diluting 1 mL into 1,000 mL DI $H_2O$. If stock solution is not 1mg/mL remake stock solution or adjust future calculations based on actual stock solution concentration.



7. Test the dose by adding the desired ratio of chlorine and ammonia-N (see examples in Table 3), to 1,000 mL DI $H_2O$. To obtain the desired ammonia-N dose, divide the free chlorine residual by the desired ratio. For example, for water with a free chlorine residual, of 2.5 mg/L and a ratio of 4.5:1, the ammonia-N dose (of the 1 mg/ml stock solution) would be 0.556 mL.



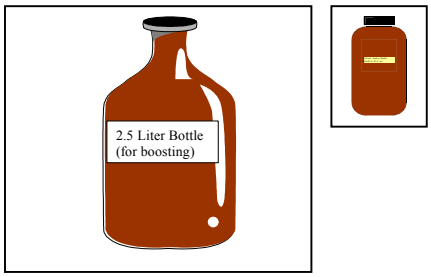
8. Test free chlorine, total chlorine, free ammonia, and total ammonia.




9. Add the amount of chlorine and ammonia necessary to obtain the desired ratio of chlorine to ammonia-N to the test water blend prepared in Step 2.

10. Stir with on a stirring plate for 15 minutes.

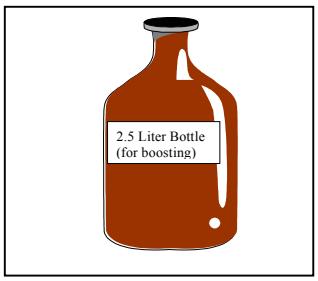
11. If the free chlorine concentration is above 0.2 mg/L, and the free ammonia-N is 0, add more ammonia.




12. Pour the test water blend into two, 2.5 L amber bottles head space free and twelve, 250 mL bottles head space free.

13. 

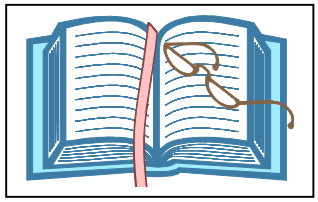
Test the water in the 250 mL bottles according to Table 4. Adjust schedule based on observations of the decay curves.

14. 


When the total chlorine residual in a 250 mL bottle drops to 1.5 mg/L or the free ammonia-N increases to 0.2 mg/L, measure the free ammonia-N, free chlorine and total chlorine in a 2.5 L bottle. Add enough chlorine into the 2.5 L bottle (from a new 1 mg/mL stock solution), to achieve the desired chlorine dose and a 4.5:1 chlorine to ammonia-N ratio. For example, if the free ammonia concentration is 0.25 mg/L $NH_3$-N, add 1.125 mg/L chlorine. The second bottle is available if needed, to repeat this step or boost another sample at a later time.

15. Stir for 15 minutes.

16. Pour the contents of the 2.5 L bottle (boosted with chlorine) into ten 250 mL bottles and repeat the tests shown in Table 4. Adjust schedule based on observations of the decay curves.

17. 

Evaluate the data to determine the decay rates before and after chlorine boosting.

| Table 1: Glassware and Reagents | | |
|---|---|---|
| | | |
| **Item** | **Size** | **Quantity** |
| Erlenmeyer Flask | 6 L | 2 |
| Carboy | 5 L | 2 |
| Carboy | 10 L | 1 |
| Volumetric Flasks | 1 L | 5 |
| Mixing Cylinder | 50 mL | 2 |
| Mixing Cylinder | 100 mL | 1 |
| Pipet | 10 mL | 2 |
| Pipet | 5 mL | 2 |
| Pipet | 1 mL | 2 |
| Amber Bottles | 2.5 L | 2 |
| Amber Bottles | 250 mL | 36 |
| Beaker | 25 mL | 2 |
| Beaker | 50 mL | 2 |
| Beaker | 250 mL | 2 |
| | | |

| **Item** | **Cat. No.** | **Qty.** | **Unit** |
|---|---|---|---|
| Total Chlorine DPD Test N' Tube | 21056-45 | 2 | Pk/50 |
| Nitri Ver 3 Reagent pillow | 21071-69 | 1 | Pk/100 |
| 10 mL Sample Cell for Free Ammonia | 24276-06 | 1 | Pk/6 |
| Free Ammonia Reagent Set | 26184-00 | 3 | 50 tests |
| NitriVer 5 Reagent pillow (optional) | 21.61-69 | 1 | Pk/100 |
| | | | |
| **Miscellaneous:** | | | |
| Alkalinity Indicator & hydrochloric or sulfuric acid | | | |
| pH standards (7 and 10), KI soln for pH probe | | | |
| Kimwipes | | | |
| Parfilm | | | |
| Tape | | | |
| Felt tip pen | | | |
| Ice pacs | | | |
| cooler or box | | | |

| Table 2: Water Blend Ratios | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | For 1 liter: Blend X and Y in number of ml shown | | | | | | | |
| **X**:Y | | **4:**1 | **5:**1 | **6:**1 | **7:**1 | **8:**1 | **9:**1 | **10:**1 |
| | **milliliters of X to add:** | **800.00** | **833.33** | **857.14** | **875.00** | **888.89** | **900.00** | **909.09** |
| | milliliters of Y to add: | 200.00 | 166.67 | 142.86 | 125.00 | 111.11 | 100.00 | 90.91 |

Table 3: Chlorine:Ammonia-N Ratios

| | | ml of 1mg/ml ammonia stock to add to 1 liter | ml of 1mg/ml chlorine stock to add to 1 liter | | | | |
|---|---|---|---|---|---|---|---|
| **14/17** | **NH3** | **NH3-N** | **4.5:1** | **4.6:1** | **4.7:1** | **4.8:1** | **4.9:1** |
| 0.824 | 0.010 | 0.008 | 0.037 | 0.038 | 0.039 | 0.040 | 0.040 |
| | 0.050 | 0.041 | 0.185 | 0.189 | 0.194 | 0.198 | 0.202 |
| | 0.100 | 0.082 | 0.371 | 0.379 | 0.387 | 0.395 | 0.404 |
| | 0.200 | 0.165 | 0.741 | 0.758 | 0.774 | 0.791 | 0.807 |
| | 0.300 | 0.247 | 1.112 | 1.136 | 1.161 | 1.186 | 1.211 |
| | 0.400 | 0.329 | 1.482 | 1.515 | 1.548 | 1.581 | 1.614 |
| | 0.500 | 0.412 | 1.853 | 1.894 | 1.935 | 1.976 | 2.018 |
| | 0.600 | 0.494 | 2.224 | 2.273 | 2.322 | 2.372 | 2.421 |
| | 0.610 | 0.502 | 2.261 | 2.311 | 2.361 | 2.411 | 2.462 |
| | 0.620 | 0.511 | 2.298 | 2.349 | 2.400 | 2.451 | **2.502** |
| | 0.630 | 0.519 | 2.335 | 2.387 | 2.438 | **2.490** | 2.542 |
| | 0.640 | 0.527 | 2.372 | 2.424 | 2.477 | 2.530 | 2.583 |
| | 0.650 | 0.535 | 2.409 | 2.462 | **2.516** | 2.569 | 2.623 |
| | 0.660 | 0.544 | 2.446 | **2.500** | 2.555 | 2.609 | 2.663 |
| | **0.675** | **0.556** | **2.501** | 2.557 | 2.613 | 2.668 | 2.724 |
| | 0.670 | 0.552 | 2.483 | 2.538 | 2.593 | 2.648 | 2.704 |
| | 0.680 | 0.560 | 2.520 | 2.576 | 2.632 | 2.688 | 2.744 |
| | 0.690 | 0.568 | 2.557 | 2.614 | 2.671 | 2.728 | 2.784 |
| | 0.700 | 0.576 | 2.594 | 2.652 | 2.709 | 2.767 | 2.825 |
| | 0.800 | 0.659 | 2.965 | 3.031 | 3.096 | 3.162 | 3.228 |
| | 0.900 | 0.741 | 3.335 | 3.409 | 3.484 | 3.558 | 3.632 |
| | 1.000 | 0.824 | 3.706 | 3.788 | 3.871 | 3.953 | 4.035 |

| **Table 4: Tests** | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | | | | |
| Sample ID | Sample Descriptor | Duplicate (D) | QA/QC (y/n) | Date | Time | Elapsed Time (Days) | Free Chlorine Residual (as $Cl_2$) | Total Chlorine Residual (as $Cl_2$) | Mono chloramine (as $NH_2Cl$-N) | Free Ammonia-N (as $NH_3$-N) | pH | Temp. ($^oC$) | Alkalinity | Nitrite | TOC | TOX |
| | 0 | | | x | | | x | x | x | x | x | x | x | x | x | x |
| | 1 | | | x | | | x | x | x | x | | | | | | |
| | 2 | | | x | | | x | x | x | x | | | | | | |
| | 3 | | | x | | | x | x | x | x | | | | | | |
| | 4 | | | x | | | x | x | x | x | | | | | | |
| | 7 | | | x | | | x | x | x | x | x | x | | | | |
| | 9 | | | x | | | x | x | x | x | | | | | | |
| | 11 | | | x | | | x | x | x | x | | | | | | |
| | 15 | | | x | | | x | x | x | x | | | | | | |
| | 17 | | | x | | | x | x | x | x | | | | | | |
| | 22 | | | x | | | x | x | x | x | x | x | x | x | | |
| | 24 | | | x | | | x | x | x | x | | | | | | |
| | 29 | | | x | | | x | x | x | x | | | | | | |
| | 36 | | | x | | | x | x | x | x | | | | | | |
| | 43 | | | x | | | x | x | x | x | | | | | | |
| | 50 | | | x | | | x | x | x | x | x | x | x | x | x | x |