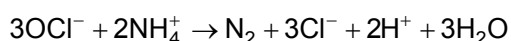
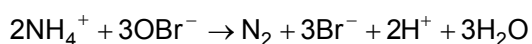
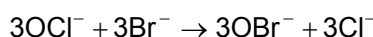


## Thermo. Titr. Application Note No. 102

<b>Title:</b>	<b>Determination of Hypochlorite by Titration with Ammonium Ion</b>
---------------	---

<b>Scope:</b>	Determination of solutions of hypochlorite
---------------	--

<b>Principle:</b>	<p>Hypochlorite ions react with bromide ions to form hypobromite ions, which in turn rapidly oxidize ammonium ions to nitrogen. Hypobromite reacts more rapidly with ammonium than hypochlorite, and is formed <i>in situ</i> (Vogel, 1961). The titration is carried out with in a solution containing bromide and bicarbonate:</p>
-------------------	--



i.e., 1 mol  $\text{NH}_4^+ \equiv 1.5$  mol  $\text{OCl}^-$

<b>Reagents:</b>	<p><i>Bromide/bicarbonate solution.</i> Dissolve 200 g A.R. KBr and 120 g A.R. <math>\text{KHCO}_3</math> in DI water and make to 1000 mL with D.I. water. Store in a brown glass bottle.</p> <p><i>Titrant. Ammonium sulfate, 0.05 mol/L (0.1 mol/L <math>\text{NH}_4^+</math>).</i> Prepare from A.R. grade ammonium sulfate, e.g., Sigma cat. no. A5132.</p>
------------------	---

<b>Method:</b>	<p><i>Basic Experimental Parameters (tiamo v.2.2.):</i></p> <table><tr><td>Titration delivery rate (mL/min.)</td><td>4</td></tr><tr><td>Titration blank (mL)</td><td>-0.0025</td></tr><tr><td>Bromide/bicarbonate solution addition (mL):</td><td>15</td></tr><tr><td>No. of exothermic endpoints</td><td>1</td></tr><tr><td>Endpoint recognition criterion (ERC)</td><td>-50</td></tr><tr><td>Delay start of titration (secs.)</td><td>5</td></tr><tr><td>Evaluation start (mL)</td><td>1</td></tr><tr><td>Damping until (mL)</td><td>0.5</td></tr></table>	Titration delivery rate (mL/min.)	4	Titration blank (mL)	-0.0025	Bromide/bicarbonate solution addition (mL):	15	No. of exothermic endpoints	1	Endpoint recognition criterion (ERC)	-50	Delay start of titration (secs.)	5	Evaluation start (mL)	1	Damping until (mL)	0.5
Titration delivery rate (mL/min.)	4																
Titration blank (mL)	-0.0025																
Bromide/bicarbonate solution addition (mL):	15																
No. of exothermic endpoints	1																
Endpoint recognition criterion (ERC)	-50																
Delay start of titration (secs.)	5																
Evaluation start (mL)	1																
Damping until (mL)	0.5																

Data smoothing factor (DSF)	40
<i>Basic Experimental Parameters (cont.):</i>	
Stirring speed (802 stirrer)	10
<i>Titration setup("semi-auto"):</i>	
6.1415.210 Titration vessel 10-90 mL	
6.1414.010 Titration vessel lid with 5 openings	
6.1909.010 Stirring propeller 96 mm	
6.2727.010 Intermediate SG sleeve	
6.1543.210 3-way stopper with anti-diffusion valve	
6.9011.020 Thermoprobe	
802 Stirrer with 804 Ti stand	
 <i>Basic Method:</i>	
Prepare an aliquot of hypochlorite solution equivalent to ~0.5-1.0 mmol OCl <sup>-</sup> . Dispense into the titration vessel, and make the fluid volume to ~15-20mL if necessary with D.I. water.	
Titrate according to the method " <i>hypochlorite by ammonium.mmet</i> ".	
 <i>Blank determination.</i>	
Prepare and titrate at least 4 hypochlorite solutions varying over a range from ~0.2 – 0.8 mmol OCl <sup>-</sup> , using the method " <i>hypochlorite by ammonium blank.mmet</i> ". Create a Common Variable " <i>Blank NH<sub>4</sub><sup>+</sup></i> " beforehand, as this method automatically saves the blank value to this CV designation.	

**Example:**

„Milton“ sterilizing solution:

NaOCl = 1.03±0.005 w/w (5 determinations)

**Calculation:**

$$\text{NaOCl}\% = \frac{((\text{EP vol} - \text{blank vol}) \times \text{NH}_4^+ \text{ mol/L} \times 74.442 \times 1.5 \times 0.1)}{\text{Sample mass, g}}$$

**Titration Plot:**

**Legend:**

Blue = solution  
temperature curve  
Black = second derivative  
curve (for endpoints)

