

## Hexavalent Chromium (Alodine 600 & Permatreat 686A):

Pipette 5 ml sample into a 250 ml Erlenmeyer Flask.

Add 50 Mls of Distilled Water.

Add 10 Mls of 10% Potassium Iodide

Add 10 Mls of Hydrochloric Acid

Let solution stand for 5 minutes.

Titrate with 0.1n Sodium Thiosulfate to a Straw Color

Add 3-5 drops of Starch Indicator

Continue Titration until Blue-Black color disappears.

### Predecessor's Calculations:

Mls of 0.1n Sodium Thiosulfate x 0.21 = oz/gal Alodine 600

Mls of 0.1n Sodium Thiosulfate x 1.25 = % by volume Permatreat 686A

### My calculations:

$$C_A \left( \frac{\text{oz}}{\text{gal}} \right) = \text{mL Thio} \left( \frac{1}{5 \text{ ml Chromic acid Sol.}} \right) \left( \frac{10^3 \text{ mL}}{L} \right) \left( \frac{0.1 \text{ mol Thio}}{10^3 \text{ mL Thio}} \right) \left( \frac{1 \text{ mol CA}}{2 \text{ mol Thio}} \right) \left( \frac{118.01 \text{ g Chromic Acid}}{1 \text{ mol Chromic Acid}} \right) \left( \frac{3.785 \text{ L}}{1 \text{ gal}} \right) \left( \frac{1 \text{ oz}}{28.349 \text{ g}} \right)$$

$$C_A \left( \frac{\text{oz}}{\text{gal}} \right) = \text{mL Thio} * 0.15 = \text{Concentration of Chromic Acid}$$

### Chemical Manufacturer's Calculations:

$$C_A \left( \frac{\text{mg}}{\text{mL}} \text{ or } \frac{\text{g}}{\text{L}} \right) = \text{mL Thio} * 1.73$$

$$C_A \left( \frac{\text{oz}}{\text{gal}} \right) = \text{mL Thio} * 1.73 * \left( \frac{3.785 \text{ L}}{1 \text{ gal}} \right) * \left( \frac{1 \text{ oz}}{28.349 \text{ g}} \right)$$

$$C_A \left( \frac{\text{oz}}{\text{gal}} \right) = \text{mL Thio} * 0.23$$

### Questions:

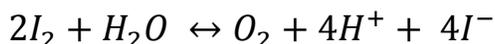
- What are the balanced equations? Is the 1 mole of Chromic Acid to 2 moles of thiosulfate the correct ratio?
- Why are these calculations different? Is it due to the fact that I would be titrating for hexavalent chromium concentration specifically? Whereas my predecessor is calculating for the amount of a chemical as a whole. Therefore if Alodine 600 is 50% chromic acid the final calculation would be twice the amount of ounces in a

gallon of solution (i.e. mL Thio \* 0.30 = Concentration of Alodine 600 in my calculation)?

- Why do both my calculation and my predecessor's calculation differ from the technical data sheet?

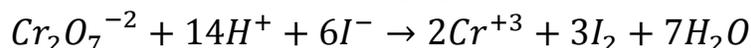
### Example Sodium Dichromate (Passivation):

I have been trying to figure out a couple things for an Iodometric titration for determination of chromic acid and sodium dichromate concentration in electroplating baths. I have found a source which outlines the mechanism by which this process works in that KI is used as an oxidizing agent giving up an electron to reduce various compounds such as Chromic Acid or Potassium Dichromate. This redox reaction liberates iodine molecules ( $I_2$ ) in an amount that is proportional to the original amount of the reduced compound. The amount of liberated iodine is then determined by titration with sodium thiosulfate using a starch indicator.

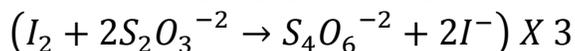


Therefore a sample is taken from a plating solution. In this case a 2 mL sample diluted with 175 mL of water. To which 15 mL of Sulfuric Acid is added to lower the pH of the solution which increases the hydrogen ion concentration in solution and forces the above reaction to the left which ensures iodine remains available.

#### Redox Reaction



#### Thiosulfate Reaction



From here 2 grams of potassium iodide (KI) is added. 6 moles of KI is required to for every mole of  $Na_2Cr_2O_7$  per the stoichiometric ratio in the balanced chemical equation. At this point the solution is titrated with a 0.1N sodium thiosulfate ( $Na_2S_2O_3$ ) reagent. It is titrated to a straw color.

The titration is not yet complete however as the yellowish, straw, color of the solutions indicates a low iodine concentration, but is not the endpoint. At this point a soluble starch indicator is added. The blue-black color of the triiodide ion in the starch is an accurate indicator of the iodine present. If starch is added into an acidic solution it would form a highly stable complex with the triiodide ion and the blue-black color could not be easily removed. The titration is then continued until the blue-black color disappears. From the above chemical reactions it is shown that for each mole of Sodium Dichromate ( $\text{NaCr}_2\text{O}_7$ ) to be reduced there is a need to 6 moles of Sodium Thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ).

$$C_A \left( \frac{g}{L} \right) = mL \text{ Thio} \left( \frac{1}{2 \text{ ml Dichromate Sol.}} \right) \left( \frac{10^3 \text{ mL}}{L} \right) \left( \frac{0.1 \text{ mol Thio}}{10^3 \text{ mL Thio}} \right) \left( \frac{1 \text{ mol Dichromate}}{6 \text{ mol Thio}} \right) \left( \frac{297.997 \text{ g Sod. Dichromate}}{1 \text{ mol Sod. Dichromate}} \right)$$

$$C_A \left( \frac{g}{L} \right) = mL \text{ Thio} * 2.483$$

$$C_A \left( \frac{\%wt}{wt} \right) = \frac{mL \text{ Thio} * 2.483}{SG_{Sol.}}$$