Hummers' Method for

Synthesis of Graphitic Oxide

- 1. Stir 100g of powdered flake graphite and 50g of sodium nitrate (NaNO $_3$) into 2.3L of 66° Baumé technical sulfuric acid (H_2SO_4) in a large jar held at 0°C in an ice bath.
- 2. Add 300g of potassium permanganate ($KMnO_4$) while maintaining "vigorous agitation". Control the rate of addition as to prevent the temperature from exceeding 20°C.
- 3. Remove from the ice bath and bring the temperature to 35±3°C for 30 minutes. At 20 minutes, the mixture should become pastey and brownish gray.
- 4. At the end of 30 minutes slowly stir $4.6L\ H_2O$ into the paste. Violent effervescence and an increase in temperature to $98^{\circ}C$ should result. The solution should remain at this temperature for 15 minutes.
- 5. Dilute further from 14L warm H_2O and treat with 3% hydrogen peroxide (H_2O_2) . The solution should turn a bright yellow upon H_2O_2 treatment.
- 6. While still warm, filter the solution.
- 7. Wash the yellowish-brown filter cake 3 times with a total of 14L warm $\ensuremath{\text{H}_{2}\text{O}}\,.$
- 8. Disperse the graphitic oxide residue in 32L $\rm H_2O$ to approximately 0.5% solids. Remaining salt impurities can be removed by treating with resinous anion and cation exchangers.
- 9. The dry form of graphitic oxide is obtained by centrifugation followed by dehydration at 40°C over phosphorus pentoxide (P_4O_{10}) in vacuo.