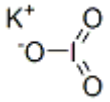
Experiment 8
Preparation of Potassium Iodate

Iodic acid, HIO_3, the only one of the halogenic acids, HXO_3, which can be isolated as the anhydrous compound, forms colourless, transparent crystals. It acts as a medium-strong acid and as a strong oxidizing agent.
Iodates, MIO_3, behave in solution in many ways like chlorates and bromates. The iodates are much more stable than chlorates and bromates, however, they can explode when mixed with organic material and carbon and heated. Iodates are also less soluble in water. They are also oxidizing agents.
The most probable conclusion is that the anion of the ordinary iodates is the simple IO_3^-.



Sodium iodate occurs in nature on Chile saltpetre, which is the main source of iodine.
Like potassium bromate, potassium iodate is occasionally used as a maturing agent in baking. Potassium iodate may be used to protect against accumulation of radioactive iodine in the thyroid by saturating the body with a stable source of iodine prior to exposure.

Part I
Preparation of Potassium Iodate

Fill a 600 mL beaker with distilled water, put it on a hot plate, and bring to boil. **Thoroughly** grind approximately 7.7-7.8 g of potassium permanganate, $KMnO_4$, to a powder using a mortar and a pestle. Weigh out 7.5 g of this powder on a weighing paper and transfer it into a 100-mL beaker. Add 30 mL of boiling water using a graduated cylinder and mix it **thoroughly** with a glass rod.
Crush approximately 3.7-3.8 g of solid iodine, I_2, in a second mortar. Weigh out 3.5 g of this powder and transfer it in very small portions into the $KMnO_4$ solution. Keep stirring with a rod. Note the original colour of this mixture. Add 4 pellets of potassium hydroxide and approximately 0.2 g of potassium iodide, KI, while stirring in the middle of the iodine addition to initiate the reaction. Rinse off the walls of the beaker with no more than 25-30 mL of boiling water. Turn off the hotplate (as it is still hot), put the beaker on it, and continue stirring until the colour of potassium permanganate completely disappears. By using a Pasteur pipet, add 1 drop of the reaction mixture onto filter paper. Your reaction is complete if you observe a drop of a brown suspension surrounded by a circle of colourless liquid. Otherwise keep stirring until this is the case. Insert a Büchner flask into a water bath with hot tap water and preheat it. Put 2-3 layers of filter paper onto the funnel. Filter the hot reaction mixture and rinse the beaker with a small amount of boiling water (try not to exceed the final volume of 100 mL of the filtrate). The filtrate should be colourless. This is an indication that the reaction between permanganate and iodide in a basic medium is complete. Transfer the filtrate into a large evaporating dish and evaporate up to 1/3 of the original volume on a hotplate (in the fume hood!). Do not forget to put 2-3 small chunks of boiling chips into it. Put the dish on ice and start stirring with a clean glass rod until crystals start forming. Cool approximately 100 mL of distilled water on an ice bath. Filter the content of the evaporating dish using a glass crucible (weighed in advance) of F porosity (consult your instructor or TA) and wash the precipitate from the evaporating dish using this cold water.
Put the crucible into a beaker and dry it in an oven at 120 $^\circ$C for about 15-20 minutes.

Part II

Prepare 10 mL of approximately 1% silver nitrate solution.

Put a small amount of potassium iodate crystals into a test tube and add the silver nitrate solution dropwise until you observe the precipitate formation.

Decant the liquid from the precipitate and add concentrated nitric acid dropwise to the precipitate using a Pasteur pipet. Record your observation. Repeat the same reactions with **your** potassium periodate.

Identification of the product by powder X-ray diffraction:

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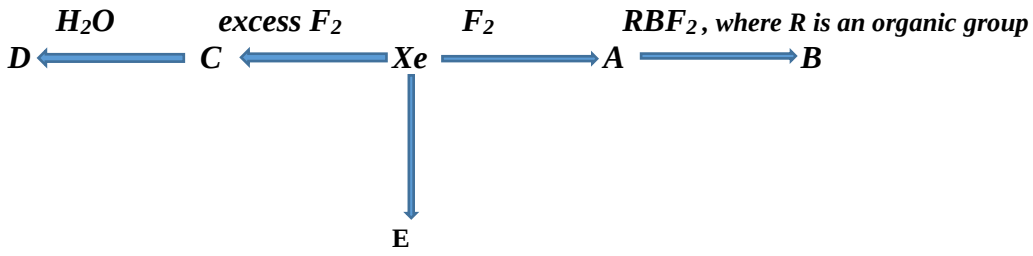
Your synthesis product needs to be identified by powder X-ray diffraction. Mount your sample on the provided sample holder. Place the sample holder in the powder X-ray diffractometer position according to your work bench label. Your sample will be measured from 10 to 90˚ in 2θ using a step width of 0.02˚. Your diffractogram will be deposited on UMLearn: CHEM2400: X-ray Data labeled with your work bench position, the weekday of your laboratory and the experiment number. You will have to identify your product using MATCH! and provide a printout of your diffractogram with the matching database entry. Clearly state if the expected product was formed. Provide comments if you do not obtain or cannot identify the expected product.

Questions

1.



2. What is the density of 53.4 % aqueous NaOH if 16.7 mL of the solution diluted to 2.00 L gives 0.169 M NaOH?