Procedure for
Oxygen Titrations

Please note—

This is analytical, not cookbook chemistry! The glassware and equipment—standard sample bottles, pipettes, stir bars, and buret tip—must be kept **SCRUPULOUSLY CLEAN**.

The glassware must be rinsed with hot water after use. **DO NOT LEAVE IT FOR SOMEONE ELSE TO DO**.

Sloppy analytical technique = nonreproducible numbers = bad grade in the class.

Dosimat Instructions

To turn on dosimat:

1. Press **FILL** button at the same time you turn on **POWER** button (red button in back).
2. Press **GO**.
3. Press **CLEAR**.
   — Display should read **DOS 0.000 ml**.

To prepare to titrate:

1. Gently turn amber bottle of thiosulfate—shake—then replace in dosimat.
2. Turn dispense speed knob to 10. Dispense 15 ml thiosulfate to flush out the buret (3-5 ml aliquots) by pressing hand control button.
3. Turn dispense speed knob to 1.
4. Press **CLEAR** button.
5. Rinse off buret tip with DIW.
6. Make sure there are no bubbles in buret or moving bubbles in line leading to buret tip.
7. Turn on stirrer to 4.

Standard Preparation

1. Fill clean standard sample bottle 3/4 full of distilled water.
2. Add 1 ml H_2SO_4, mix well.
3. Slowly add 1 ml NaOh-NaI, mix well.
   — If sample is not clear, discard and start again.
4. Using 10 mlA volumetric pipette, add 10 ml KIO_3 standard.
   — Always shake the standard before pipetting.
   — Pour ~20 ml into small plastic beaker.
   — Draw standard from the small beaker.
   — Remove the pipette from the beaker. **NEVER DRAIN LIQUID BACK INTO STANDARD BOTTLE OR BEAKER**.
   — Wipe down tip of pipette with kimwipe.
   — Dispense into sample bottle. Do not put tip of pipette against wall of sample bottle—this will cause excess standard to be delivered from pipette.
5. Position sample bottle on stirrer; make sure buret tip is under the surface of the sample.
6. Make sure dosimat reads **0.000 ml** (press **CLEAR** to zero).
7. Titrate sample by dispensing thiosulfate into the sample.
   — Use the thumb-button gizmo to dispense thiosulfate.
8. When the sample is light yellow in color, add 1 ml starch indicator.
9. Titrate to endpoint.
   — Endpoint is when all color is gone; watch vortex in upper half of bottle.
   — The endpoint is subtle—the difference between clear and sparkling clear.
10. Record endpoint.
11. Remove sample bottle; dispense a few drops of thiosulfate through buret tip to flush out any sample residue.
12. Rinse down buret tip with DIW.
13. Press **CLEAR** to zero dosimat.
14. Run at least 3 standards; at least 2 out of 3 should agree ± .001 ml.
15. After analysis, rinse bottles with hot water and store filled with distilled water.

Blank

The blank is a correction factor. The reagents may add a contamination to the standard and sample measurements. This contamination may be due to impurities in the crystalline form of the reagent or because the liquid reagent was contaminated by "something" (seawater, sunlight, another chemical) while in use aboard ship.

To prepare a blank:

1. Fill a standard sample bottle 3/4 full of distilled water.
2. Add 1 ml H_2SO_4, mix well.
3. Slowly add 1 ml NaOH-NaI, mix well.
4. Add 1 ml $MnCL_2$, mix well.
5. Using automatic pipette, add 1.0 ml KIO_3 standard.
6. Add starch immediately (because the sample is light yellow).
7. Position sample bottle on stirrer; make sure buret tip is under the surface of the sample. (Titrate slowly; remember this is only 1/10 as strong as the standard.)
8. Make sure dosimat reads **0.000 ml** (press **CLEAR** to zero).
9. Titrate sample by dispensing thiosulfate into the sample.
   — Use the thumb-button gizmo to dispense thiosulfate.
10. Titrate to endpoint.
    — Endpoint is when all color is gone; watch vortex in upper half of bottle.
    — The endpoint is subtle—the difference between clear and sparkling clear.
11. Record endpoint #1.
12. Press **CLEAR** button.
13. Add 1.0 ml more of KIO_3 standard.
14. Titrate to the endpoint #2.
15. (Endpoint #1) – (Endpoint #2) = blank correction factor

Translation

Endpoint #1 (in ml) = volume of thiosulfate needed to titrate the first 1 ml KIO_3 + reagents.

Endpoint #2 (in ml) = volume of thiosulfate needed to titrate the second 1 mlKIO_3.

Therefore (Endpoint #1) – (Endpoint #2) = volume of thiosulfate needed to titrate reagents.

This value may be negative or positive or zero.

Titrating Samples

1. Carefully remove cap, rinse glass stopper.
2. Add clean stir bar.
3. Add 1 ml H_2SO_4, mix well.
4. Position sample bottle on stirrer; make sure buret tip is under the surface of the sample.
5. Make sure dosimat reads **0.000 ml** (press **CLEAR** to zero).
6. Titrate sample by dispensing thiosulfate into the sample.
   — Use the thumb button gizmo to dispense thiosulfate.
7. When the sample is light yellow in color, add 1 ml starch indicator.
8. Titrate to endpoint.
   — Endpoint is when all color is gone; watch vortex in upper half of bottle.
   — The endpoint is subtle—the difference between clear and sparkling clear.
9. Record endpoint.
10. Remove sample bottle; dispense a few drops of thiosulfate through buret tip to flush out any sample residue.
11. Rinse down buret tip with DIW.
12. Press **CLEAR** to zero dosimat.