


Pollution lab 2

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Determination of dissolved oxygen (Test procedure For Winkler method)

Test procedure For Winkler method

1- Sample collection

- ▶ When collecting your water sample, here are some important guidelines:
- ▶ Collect the water sample away from the bank and below the water surface level.
- ▶ Be careful not to get any air bubbles in the sample during collocation, as it may result in false high reading.
- ▶ • Allow the water to fill the dissolved oxygen (DO) sample bottle from bottom to top.
- ▶ • Put a lid on the bottle while in under water.

Test the dissolved oxygen (DO) level immediately, As the biological activity in the sample and exposure to air can quickly change the dissolved oxygen (DO) level.

Test procedure For Winkler method

2- Laboratory work

- ▶ 1. Add gently, and just below the surface, 2 ml of MnSO_4 reagent and 2 ml of the mixed NaOH-KI reagent. Do not mix the pipettes between reagents, and do not mouth pipette the reagents. Carefully stopper the bottle without introducing any air bubbles and mix vigorously by inverting the bottle repeatedly.
- ▶ 2. Allow the precipitate to settle, then shake vigorously again and allow precipitate to settle to at least the bottom third of the bottle. If the rest of the analysis has to be delayed, the samples will normally keep quite well in this condition.

Test procedure For Winkler method

2- Laboratory work

- ▶ **3.** Add 2 ml of concentrated H_2SO_4 , inserting the tip of the pipette just below the surface of the sample. Carefully re-stopper the bottle, avoiding bubbles, and shake until all the precipitate has dissolved. Samples can also be stored in this condition if protected from light, but the free iodine has a high vapor pressure and tends to escape even from well-capped bottles. Delay of further analysis beyond 8 hours is not desirable.
- ▶ **4.** Measure 100 ml of the sample with a volumetric pipette and transfer to a 250 ml flask. For accurate delivery, the tip of the volumetric pipette should be touched to the side of the flask during delivery.

Test procedure For Winkler method

2- Laboratory work

- ▶ **5.** Using a 50 ml burette filled with 0.025N standardized sodium thiosulfate solution, titrate with mixing until a pale straw
- ▶ **6.** Add 2 ml of stabilized starch mixture, mix to get a uniform blue color, and continue titrating carefully but rapidly to a colorless end-point. The blue color should return if the sample is left standing from 15 to 20 seconds, and can be ignored. If the blue color does not return, the end-point has been overshoot. Record the volume of titrant used in ml.

Test procedure For Winkler method

2- Laboratory work

- ▶ 7. Calculate the concentration of DO in the sample using the following formula:

- ▶
$$\text{DO (mg/L)} = \frac{(\text{mL titrant} \times \text{normality of titrant} \times 8000)}{\text{Equivalent volume of sample titrated}}$$

Test procedure For Winkler method

Azide-Winkler Method

