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QUALITY CONTROL/QUALITY ASSURANCE DOCUMENTATION

Title: Measuring Dissolved Oxygen by the “Winkler-Azide” Method
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REVISION HISTORY

Revision	Change Description	Date	Authorization
1	Reviewed by AL	11/16/21	AL

ACKOWLEDGEMENTS

(Place to acknowledge peer reviewer)

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1.0 SCOPE AND APPLICABILITY

This document presents the North Dakota Department of Environmental Quality, Division of Water Quality's (DWQ) Standard Operating Procedure (SOP) for measuring dissolved oxygen (DO) by the "Winkler-Azide" method. This SOP applies to all DWQ field staff, non-DWQ cooperators, and citizen volunteers.

2.0 SUMMARY OF METHODS

Measurements made using the "Winkler-Azide" method are considered the most accurate measurement of dissolved oxygen. The "Winkler-Azide" method can be used for making direct measurements of dissolved oxygen in surface waters, field calibrating a dissolved oxygen meter, and performing regularly scheduled dissolved oxygen meter calibrations. The following are the procedures for measuring dissolved oxygen by the "Winkler-Azide method. A detailed description of the method can be found in Standard Methods, (17th Edition, APHA, 1989).

3.0 HEALTH AND SAFETY WARNING

In field situations, personnel should take appropriate precautions when operating watercraft and working on, in, or around water. All boats used for sampling should be equipped with safety equipment such as personal flotation devices (PFD's), oars, air horn, etc. North Dakota's boating laws and rules shall be followed by all field personnel.

In the laboratory setting, appropriate attire should be dawned such as protective eyewear, gloves, and aprons in order to minimize any potential splatter associated with this lab process.

4.0 CAUTIONS

Care should be taken when collecting the sample and when filling the BOD bottle so as to not introduce air bubbles into the sample.

5.0 INTERFERENCES

The most common interferences to the "Winkler-Azide" method are oxidizing and reducing agents, nitrate ion, ferrous iron, and organic matter.

6.0 PERSONNEL QUALIFICATIONS/RESPONSIBILITIES

All personnel taking field measurements must read this SOP annually and acknowledge they have done so via a signature page (see Appendix B). New field personnel must also demonstrate successful performance of the method. The signature page will be signed by both trainee and trainer to confirm that training was successfully completed and that new personnel is competent in carrying out this SOP. The signature page will be kept on-file at DWQ along with the official hard copy of this SOP.

7.0 EQUIPMENT AND SUPPLIES

- 300 ml BOD bottles
- 300 ml manganous sulfate solution
- 300 ml alkaline-iodide-azide reagent
- Concentrated sulfuric acid
- Graduated cylinder capable of measuring 203 ml
- Titration equipment capable of measuring to 0.1 ml
- 0.025 M sodium thiosulfate solution
- Starch indicator solution
- Kemmerer or Van Dorn type sampler

8.0 PROCEDURE

1. Obtain a sample by completely filling a 300 ml BOD bottle. The bottle is filled slowly taking care to exclude air bubbles. Note: When measuring dissolved oxygen for calibration purposes two samples should be collected at the same time and place. The results of the two should agree within +/- 0.2 mg/l. If not, repeat the procedure.
2. Insert the glass stopper to remove excess water trapped in the neck of the bottle.
3. Add 2 ml of manganous sulfate.
4. Add 2 ml of alkaline iodide-azide
5. Stopper the bottle, taking care to exclude air bubbles.
6. Mix well by inverting and righting the bottle rapidly for 1 minute, at which time a flocculent will have formed.
7. Let the flocculent settle until approximately 1/3 of the bottle has cleared, then mix again.
8. Let the flocculent settle until approximately 2/3 of the bottle has cleared.

9. Add 2 ml sulfuric acid, stopper, and mix. Note: Take care when adding sulfuric acid as any spillage can cause severe burns. If any acid is spilled it should be flushed immediately with water.

10. At this point the oxygen within the sample is fixed and the sample may be stored in the dark for up to 8 hours.

11. Using the graduated cylinder, transfer 203 ml of the sample to the titration vessel.

12. While gently swirling sample, titrate with the sodium thiosulfate solution to a pale straw color.

13. Add 2 ml starch indicator solution. Sample will turn blue.

14. Continue to titrate and swirl the sample until the first disappearance of blue color.

15. Record to nearest 0.1 ml the volume of titrant used as mg/l dissolved oxygen in the sample.

9.0 DATA AND RECORDS MANAGEMENT

Sample information will be recorded on the field form (Appendix A). Once personnel reach the office, data recorded on the field form are entered into the DWQ Sample Identification Database (SID). Field notes should be used to record any quality control activity performed such as measurements taken by more than one sampler, or to record any sampling conditions that may have interfered with the reading such as high winds/wave action, cattle in water, observed flow, water surface, water clarity, water color, water odor, visual algae cover, number of dead fish, present weather, estimated inches of rain fall in past 72 hours, and any comments. Field forms and notes should be stored in the appropriate project folder at DWQ.

10.0 QUALITY ASSURANCE AND QUALITY CONTROL

The meter(s) should be calibrated before sampling trip following the manufacturer's instructions and the calibrations should be recorded. Additionally, temperature probes will be checked annually against a certified NIST thermometer.

11.0 REFERENCES

Standard Methods.
17th Edition, APHA, 1989.

APPENDIX A

Field Reporting Forms

River and Stream Sampling Field Log
North Dakota Department of Environmental Quality
Division of Water Quality - Watershed Management Program
Telephone: 701-328-6140
Fax: 701-328-6280

Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date:___/___/_____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date:___/___/_____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date:___/___/_____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date:___/___/_____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date:___/___/_____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date:___/___/_____	Temperature	DO	
		Time: :	SC	pH	
Sample #:		Site ID:	Site Description:		Comments:
Dup	Blk	Date:___/___/_____	Temperature	DO	
		Time: :	SC	pH	

Lake Profile Field Log
North Dakota Department of Environmental Quality
Division of Water Quality – Watershed Management Program
Telephone: 701-328-6140
Fax: 701-328-6280

Project Code:		Project Name:	
Site Identification:		Sampler(s):	
Site Description:			
Date: / /	Time: :	Ambient Temp:	Wind Speed: (mph)
Wind Direction:	% Cloud Cover:	Secchi Disk: (m)	Baro: (mm/Hg)
Chlorophyll-a:	Phytoplankton:	Initial DO:	Final DO:
Sample Depths: _____ Meters, _____ Meters, _____ Meters, _____ Meters			
Comments:			

Depth (m)	Temp (c)	DO (Mg/L)	pH	Specific Conduct.	Comments

Wadable Wetland Field Sampling Form
North Dakota Department of Environmental Quality
Division of Water Quality – Watershed Management Program
Telephone: 701-328-6140
Fax: 701-328-6280

Site ID:	Wetland Name or description:			County:
Observer:			Date:	Time:
Aquatic Zone Description:				
Ambient Temp:		Wind Speed:		Wind Direction:
Cloud Cover % of 100	Secchi (m)	chlorophyll-a=(y / n)	Phytoplankton=(y / n)	
Comments:				
Water Chemistry Taken At	Meters,	Meters,	Meters,	Meters,

Depth (m) or Location	Temp. (C)	D.O. (mg/L)	pH	Conductivity (umhos/cm)	Comments

Site Drawing:

APPENDIX B

SOP Acknowledgement and Training Form

SOP Acknowledgement and Training Form

This SOP must be read, and this form signed annually. This form must be kept with the latest version of the SOP.

Document Title:	
Document Revision Number:	
Document Revision Date:	

Please sign below in accordance with the following statement:
“I have read and understand the above referenced document. I agree to perform the procedures described in this SOP in accordance with the document until such time that it is superseded by a more recent approved revision.”

Printed Name	Signature	Date

SOP Acknowledgement and Training Form (cont.)

Trainee: Sign below to acknowledge that training on this SOP was received, understood, and all questions/concerns were addressed by the trainer.

Trainer: Sign below to acknowledge that training on this SOP was completed for the individual listed and that training is competent to perform the procedures described within.

Date of Training	Trainee Printed Name	Trainee Signature	Trainer Printed Name	Trainer Signature