

# Dissolved Oxygen

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Arizona Department of Health Services  
AZPDES/APP Permit Field Method Training  
Fall 2016



# Approved Methods

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- Standard Methods – 22<sup>nd</sup> edition
  - SM 4500-O B (2011)– Iodometric Method
  - SM 4500-O C (2011)– Azide Modification
  - SM 4500-O D (2011)– Permanganate Mod.
  - SM 4500-O E (2011)– Alum Flocculation Mod.
  - SM 4500-O F (2011)– Copper Sulfate-Sulfamic Acid Flocculation Mod.
  - SM 4500-O G (2011)– Membrane Electrode\*
- HACH 10360 – Luminescence\*

# Preservation & Holding Times

## 40 CFR § 136.3 (Table II)

38. Nitrate	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	48 hours.
39. Nitrate-nitrite	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$ , $\text{H}_2\text{SO}_4$ to pH <2	28 days.
40. Nitrite	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	48 hours.
41. Oil and grease	G	Cool to $\leq 6^{\circ}\text{C}^{18}$ , HCl or $\text{H}_2\text{SO}_4$ to pH <2	28 days.
42. Organic Carbon	P, FP, G	Cool to $\leq 6^{\circ}\text{C}^{18}$ , HCl, $\text{H}_2\text{SO}_4$ , or $\text{H}_3\text{PO}_4$ to pH <2	28 days.
44. Orthophosphate	P, FP, G	Cool, to $\leq 6^{\circ}\text{C}^{18, 24}$	Filter within 15 minutes; Analyze within 48 hours.
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes.
47. Winkler	G, Bottle and top	Fix on site and store in dark	8 hours.
48. Phenols	G	Cool, $\leq 6^{\circ}\text{C}^{18}$ , $\text{H}_2\text{SO}_4$ to pH <2	28 days.
49. Phosphorous (elemental)	G	Cool, $\leq 6^{\circ}\text{C}^{18}$	48 hours.
50. Phosphorous, total	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$ , $\text{H}_2\text{SO}_4$ to pH <2	28 days.
53. Residue, total	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	7 days.
54. Residue, Filterable	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	7 days.
55. Residue, Nonfilterable (TSS)	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	7 days.
56. Residue, Settleable	P, FP, G	Cool, $\leq 6^{\circ}\text{C}^{18}$	48 hours.

# SM 4500-O G

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- Membrane electrode method
  - Robust method for field, continuous, in situ, and lab monitoring
  - Oxygen sensitive membrane electrodes
    - Most are linearly proportional to the concentration of molecular oxygen which allows easy conversion to concentration units
  - Be aware of temperature effects
    - Some electrode manufacturers provide charts showing effects
    - Check frequently to verify temperature correction data



# SM 4500-O G

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## ○ Calibration

- Manufacturer procedure
  - Follow instructions/notes provided with instrument and/or electrodes
- Common methods
  - Reading against air
    - Temperature
    - Barometric
  - Reading a known DO concentration and a sample with zero DO



# General Items

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## ○ Sampling

- Minimize contact with air
- Do not agitate
- Both change gaseous content

## ○ Duplicates

- Analyze at least one duplicate for each matrix type daily or with each batch of 20 or fewer samples.
- Two samples taken at the same time from one location



TABLE 4020-I. CONT

Section	Method Blank	LFB*	LFM† & LFMD‡	Other	Section	Method Blank	LFB*	LFM† & LFMD‡	Other
4500-IO <sub>3</sub> <sup>-</sup> B	X	X	X	3	4500-O B	-	-	-	2,6
4500-N B	X	X	X	3	4500-P C	X	X	X	3
4500-N C	X	X	X	3	4500-P D	X	X	X	3
4500-N D	X	X	X	3	4500-P E	X	X	X	3
4500-NH <sub>3</sub> C	X	X	X	3	4500-P F	X	X	X	3
4500-NH <sub>3</sub> D	X	X	X	3	4500-P G	X	X	X	3
4500-NH <sub>3</sub> E	X	X	X	3	4500-P H	X	X	X	3
4500-NH <sub>3</sub> F	X	X	X	3	4500-P I	X	X	X	3
4500-NH <sub>3</sub> G	X	X	X	3	4500-P J	X	X	X	3
4500-NH <sub>3</sub> H	X	X	X	3	4500-KMnO <sub>4</sub> B	X	X	X	3
4500-NO <sub>2</sub> <sup>-</sup> B	X	X	X	3	4500-SiO <sub>2</sub> C	X	X	X	3
4500-NO <sub>3</sub> <sup>-</sup> B	-	-	-	3	4500-SiO <sub>2</sub> D	X	X	X	3
4500-NO <sub>3</sub> <sup>-</sup> C	X	X	X	3	4500-SiO <sub>2</sub> E	X	X	X	3
4500-NO <sub>3</sub> <sup>-</sup> D	X	X	X	3	4500-SiO <sub>2</sub> F	X	X	X	3
4500-NO <sub>3</sub> <sup>-</sup> E	X	X	X	3	4500-S <sup>2-</sup> D	X	X	X	3
4500-NO <sub>3</sub> <sup>-</sup> F	X	X	X	3	4500-S <sup>2-</sup> E	X	X	X	3
4500-NO <sub>3</sub> <sup>-</sup> H	X	X	X	3	4500-S <sup>2-</sup> F	X	X	X	3
4500-NO <sub>3</sub> <sup>-</sup> I	X	X	X	3	4500-S <sup>2-</sup> G	X	X	X	3
4500-N <sub>org</sub> B	X	X	X	3	4500-S <sup>2-</sup> I	X	X	X	3
4500-N <sub>org</sub> C	X	X	X	3	4500-S <sup>2-</sup> J	X	X	X	3
4500-N <sub>org</sub> D	X	X	X	3	4500-SO <sub>3</sub> <sup>2-</sup> B	X	X	X	3
4500-O C	-	-	-	2,6	4500-SO <sub>3</sub> <sup>2-</sup> C	X	X	X	3
4500-O E	-	-	-	2,6	4500-SO <sub>4</sub> <sup>2-</sup> C	X	X	X	3
4500-O F	-	-	-	2,6	4500-SO <sub>4</sub> <sup>2-</sup> D	X	X	X	3
4500-O <sub>3</sub> B	X	-	-	2	4500-SO <sub>4</sub> <sup>2-</sup> E	X	X	X	3
4500-O G	-	-	-	2,6	4500-SO <sub>4</sub> <sup>2-</sup> F	X	X	X	3
					4500-SO <sub>4</sub> <sup>2-</sup> G	X	X	X	3

\* Laboratory-fortified blank.

† Laboratory-fortified matrix.

‡ Laboratory-fortified matrix duplicate.

X indicates that a QC type is mandatory for the method.

1. Additional QC guidelines in method.

2. Duplicates of the sample will be run. ←

3. Refer to 4020B for further QC requirements.

4. Compare to results from Section 4500-CO<sub>2</sub>, 4500-CO<sub>2</sub>.2.D.

5. Additional QC check with pH standard whose value is bracketed by calibration standards.

6. Zero check with zero oxygen sample. ←

This table is not comprehensive; refer to the specific method and 4020B for further details.

# SM 4500-O G

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- Check the electrode at a minimum for:
  - Storage solution, if used – correct type, not dried, filled
  - Look for cracks, scratches
  - Do not use paper towels
  - If membrane needs replacement
  - Ensure when using to read samples, no trapped air bubbles under membrane





# Oxygen, Dissolved

DOC316.53.01243

## Direct Measurement Method<sup>1</sup>

Method 10360

0.1 to 20.0 mg/L (or 1 to 200% saturation) O<sub>2</sub>

LDO probe

**Scope and application:** For water, wastewater and process water applications.

<sup>1</sup> Luminescent dissolved oxygen is USEPA approved for dissolved oxygen measurements in wastewater treatment process (e.g., aeration and biological nutrient basins, effluent outfalls and receiving water). Refer to USEPA 40CFR Part 136.3.



## Test preparation

### Instrument-specific information

This procedure is applicable to the meters and probes that are shown in [Table 1](#). Procedures for other meters and probes can be different.

**Table 1 Instrument-specific information**

Meter	Probe
HQ30d portable single input, multi-parameter HQ40d portable dual input, multi-parameter HQ430d benchtop single input, multi-parameter HQ440d benchtop dual input, multi-parameter	IntelliCAL LDO101 LDO

### Before starting

Refer to the meter documentation for meter settings and operation. Refer to probe documentation for probe preparation, maintenance and storage information.

Prepare the probe before initial use. Refer to probe documentation.

When an IntelliCAL™ probe is connected to an HQd meter, the meter automatically identifies the measurement parameter and is prepared for use.

The IntelliCAL LDO101 probes automatically adjust for barometric pressure, elevation and temperature.

Do not touch the probe cap with a hand, fingers or any surface that can scratch the cap.

Prepare the probe before initial use. Refer to probe documentation.

Condition the probe before use. To condition the probe, put the probe in 100 mL of tap water for 30 minutes before use.

For probes that are continuously in aqueous solutions, condition the sensor cap for 72 hours.

# Field Video Demonstration

