EPA Region 9 Supplemental Guidance to Hach Method 10360
Luminescence Measurement of Dissolved Oxygen in Water and Wastewater
Revision 1.0
April 2008

1.0 Scope and Application

1.1 This supplemental guidance document is applicable to the United States Environmental Protection Agency Region 9 states of Arizona, California, Hawaii, and Nevada when using Hach Method 10360 (Rev. 1.1, January 2006) for the measurement of dissolved oxygen (DO) in wastewater effluent outfalls and in biochemical oxygen demand (BOD) determinations.

1.2 The guidance document provides clarification to the Scope and Application, Method Detection Limit, Quality Control, Method Performance, Table and Glossary of Definitions and Purposes sections of the method.

1.3 The numeric section changes described in this document correspond to the numbering system of the method. Specifically, sections 1, 6, 8, 12, 16, and 17 of Hach Method 10360, describing preparation of reference water samples for demonstration of Method Detection Limit (MDL), Minimum Limit (ML), Initial Precision and Recovery (IPR), are being modified.

1.4 For a complete understanding of the scope, application and performance of Hach Method 10360, the user is referred to the Hach Validation Study available for download at www.Hach.com.

1.5 The supplemental guidance document is valid until the time when DO reference water becomes commercially available or until Hach Method 10360 is promulgated and published at Title 40 of the Code of Federal Regulations, which ever comes first.

2.0 Changes to Section 1.0 (Scope and Application)

2.1 The MDL of 0.05 mg/L and ML of 0.20 mg/L stated in section 1.5 of the method are substantially below the regulatory reporting limit for DO in treated effluent and DO in the determination of BOD (1.0 to approximately 10 mg/L). Additionally, DO reference water at a concentration to perform the MDL and ML verification is not commercially available. Until DO reference water becomes commercially available, the laboratory demonstration to achieve the MDL and ML is not required for NPDES regulatory reporting of DO.

3.0 Changes to Section 6.3 (Method Detection Limit)

3.1 Section 6.2 describes the preparation of calibration verification, initial precision and recovery, and on-going precision and recovery samples. If calibration verification, initial precision and recover, and on-going precision and recovery are for demonstration with BOD samples, the laboratory may choose as an alternative, to use BOD dilution water. Delete section 6.2 and replace with the following: Calibration Verification, Initial Precision and Recovery, and On-going Precision and Recovery - Add approximately 1500 mL of organic-free water to a 2-L beaker. If calibration verification, initial precision and recover, on on-going precision and recovery are for demonstration with BOD samples, the laboratory may choose as an alternative, to use BOD dilution water.

3.2 Section 6.3 and its sub-sections 6.3.1 and 6.3.2 describe the preparation of DO reference water. Dissolved oxygen reference water at a concentration required to perform the MDL is not commercially available and is not technically feasible for the laboratory to prepare. Until DO reference water becomes commercially available, the laboratory demonstration to achieve the MDL is not required for NPDES regulatory reporting of DO.

4.0 Changes to Section 8.0 (Quality Control)

4.1 Sections 8.2.1 and 8.2.2 of the method describe the calculation of MDL and ML. DO reference water at a concentration required to perform and calculate the MDL and ML is not commercially available and is not technically feasible for the laboratory to prepare, Until DO reference water becomes commercially available, the laboratory demonstration to achieve the MDL and ML is not required for NPDES regulatory reporting of DO.

4.2 Section 8.3 and 8.4 of the method describes the preparation of DO reference water for demonstration of calibration verification (CV) and on-going precision and recovery (OPR). The DO

reference water concentration for the CV and OPR shall be prepared at air-saturated water under standard laboratory temperature and barometric pressure (ambient) conditions. The greatest bias in the LDO technology is at DO concentrations of air saturation. Therefore, performing an IPR and OPR at air saturation best demonstrates performance of the method.

4.2.1 Delete section 8.3.1 of the method and replace with the following: Prepare a calibration verification standard (Section 6.2) at a DO concentration representative of air-saturation under standard laboratory temperature and pressure (ambient) conditions with each analytical batch. Analyze according to the procedure beginning in Section 10 and compare the recovery results to those in Table 3. Actual average recovery and standard deviation should be within the specifications in Table 1.

4.2.2 Delete section 8.4.1 of the method and replace with the following: Prepare a precision and recovery standard (Section 6.2) at a DO concentration representative of air-saturation under standard laboratory temperature and pressure (ambient) conditions with each analytical batch according to the procedure beginning in Section 10.

4.2.3 Section 8.4.3 states the accuracy is 85% to 105%. This is incorrect. The accuracy is 95% to 105%. Delete section 8.4.3 and replace with the following: The laboratory should add results that pass the specification in Table 1 to IPR and previous OPR data and update QC charts to form a graphic representation of continued laboratory performance. The laboratory should also develop a statement of laboratory data quality for DO by calculating the average percent recovery and the standard deviation of the percent recovery. The criterion that must be met is 95% to 105% recovery.

5.0 Changes to Section 12.0 (Method Performance)

5.1 The MDL and ML Acceptance Criterion of 0.05 mg/L and 0.20 mg/L respectively, is substantially below the regulatory reporting limit for DO in treated effluent and DO in the determination of BOD (1.0 to approximately 10 mg/L). Additionally, DO reference water at a concentration to perform the MDL and ML verification is not commercially available. Until DO reference water becomes commercially available, the laboratory demonstration to achieve the MDL and ML is not required for NPDES regulatory reporting of DO.

6.0 Changes to Section 16.0 (Tables)

6.1 The Initial Precision and Recovery Method Performance (IPR) lists recovery and precision criterion for a low and high range of DO concentration, DO reference water at a concentration to perform the low IPR verification is not commercially available. Until DO reference water becomes commercially available, the laboratory demonstration to achieve the low range IPR is not required for NPDES regulatory reporting of DO. Delete Table 1. Initial Precision and Recovery Method Performance and replace with the following:

IPR DO Conc. (mg/L)	97.5% Lower Limit of Recovery (%)	97.5% Upper Limit of Recovery (%)	95% Upper Limit of Precision (%)
7.22 – 9.23	95.8	104.8	1.26

6.2 The MDL and ML Acceptance Criterion of 0.05 mg/L and 0.20 mg/L respectively, is substantially below the regulatory reporting limit for DO in treated effluent and DO in the determination of BOD (1.0 to approximately 10 mg/L). Additionally, DO reference water at a concentration to perform the MDL and ML verification is not commercially available. Until DO reference water becomes commercially available, the laboratory demonstration to achieve the MDL and ML is not required for NPDES regulatory reporting of DO.

7.0 Changes to Section 17.0 (Glossary of Definitions and Purposes)

7.1 Until such time that DO reference water becomes commercially available, the MDL and ML in this Supplemental Guidance Document are not a requirement Hach Method 10360 (Rev. 1.1, January 2006) for the measurement of dissolved oxygen (DO) in wastewater effluent outfalls and in biochemical oxygen demand (BOD) determinations. The definitions, acronyms, and abbreviations of MDL and ML at sections 17.2.5 and 17.2.6 are simlilary not applicable.