



Standard Operating Protocol for Verification of Installation and Calibration of UV-Vis Dual Beam Scanning & Multipoint calibration mechanism technology based OCEMS (Effluent) used in Sewage Treatment Plants (STPs)

Version 1.0

This protocol provides general guidance for verification of Online Continuous Effluent Monitoring System (OCEMS) installed at Sewage Treatment Plants (STPs) with step-wise approach for verification of installation and initial calibration by instrument installer, later validation of calibration by operator and performance check by the State Pollution Control Boards (SPCBs)/Pollution Control Committees (PCCs), so as to ensure that reliable data is generated by OCEMS.

1. Verification of OCEMS Installation

- (i) **Pre-requisites**-OCEMS installation should be planned w.r.t. availability of location, uninterrupted stabilized power supply, protection from harsh environmental conditions, security, accessibility, safety of access to sensors and data display board, data transmission with remote or manual calibration procedure etc. These are pre-requisites before installation of OCEMS.
- (ii) **Continuous Operation**: The purpose of installation of online analyser is to get continuous effluent monitoring data, hence, in no case the system shall be switched off without prior intimation to the concerned regulators.
- (iii) **In-Situ location**: OCEMS must be installed at the Final Outlet/stable outlet channel with least water turbulence. It should always be placed at the treated water chamber overflowing in to final outlet.
- (iv) The sensor must always remain submerged in effluent and whenever (during maintenance and calibration) sensor is taken out from the effluent, alarm should be generated and communicated to SPCB/CPCB server automatically.
- (v) **Extractive location**: The sample must be drawn through a pump from the same location as mentioned above with little over pressure to maintain the sample supply continuously. In case the sample is not available, system should generate automated alarm communicated to CPCB/SPCB server.

2. Calibration of OCEMS:

STP Operators may deploy different makes of instruments based on different technologies measuring effluent quality in terms of pH, BOD, COD, and TSS, therefore, operation and calibration mechanism of such instruments will differ. This document covers calibration procedure for

commonly used technologies for effluent monitoring, available as on date, that is UV-Vis principle based system for BOD, COD & TSS measurement having dual beam scanning and Multipoint calibration mechanism.

Complete calibration of OCEMS requires following checks, calibrations & validations:

- (i) Function Check
- (ii) Basic Installation & Calibration
- (iii) Continuous Validation
- (iv) Performance Check

The calibration shall be considered complete, when process no. (i), (ii), (iii) and (iv) have been completed successfully and all the conditions of protocol are found to be within permissible ranges/limits mentioned in this document.

Function checks, Installation & calibration check is the responsibility of the firm or its authorized representative installing OCEMS. Ensuring continuous validation of OCEMS is the responsibility of the operator of STPs (to be verified by SPCBs/PCCs). Performance check of OCEMS is the responsibility of SPCBs/PCCs to verify the performance of plants against stipulated standards.

2.1 OCEMS Function check

The purpose of this exercise is to cross verify the operation of basic electronics involved in the system. When required, this data should be shared through email to SPCBs/PCCs. Records of this function check should be available in the plant.

Parameter-wise function check process is below:

- pH: Calibrate pH meter with Certified (having international traceability) Buffer solutions of pH 4, 7, 9.2 & 10. Perform at-least two point calibration within the expected range of the pH in the plant. For example, if pH is expected to be 7.8 then perform two points calibration with pH 7 and 9.2.
- BOD: Not possible
- COD: Use KHP Certified Reference Material (CRM)

Step 1: Prepare different dilutions using Potassium Hydrogen Phthalate (KHP) with stock solution 1000ppm and check performance of instrument in respective dilutions. Calibrate optics of the system with the procedure detailed below.

Step 2: Prepare stock standard (1000ppm equivalent COD) in laboratory and store it in refrigerator. Stock is stable upto six months, if stored in refrigerator. Hence, as a safety margin, do not use stock older than five months in calibration and also do not use stocks, if exposed in environment because of any reason.

Step 3: Prepare working solution of nearly 1/3rd, 1/2, 2/3rd and 2 times of working range (i.e. COD levels expected in a plant) and perform the functional test on site within 24 hours.

Step 4: Verify the results and if not found within 5% of the range, perform functional check again

Step 5: Submit middle dilution (of the range in use at plant) as sample for parallel COD measurements in lab along-with the samples.

TSS: Use Formazine solution. Care to be taken in handling solution.

Frequency of function test is every 15 days. STPs may engage the firms providing services of O & M.

2.2 Basic Installation and Calibration of OCEMS

The purpose of this exercise is to ensure installation of the system in a plant and collect calibration data points for better matrix matching and to authenticate the range of measurement coverage. The data collected should be shared through email to SPCBs/PCCs or CPCB. Copy of this basic or initial calibration should be available at any point of time in the plant. If such exercise was not done earlier, the same should be completed and records be maintained. Basic calibration test should be repeated when two consecutive validation tests or performance tests fail and results fall beyond the “criteria of outliers” given in this document.

Step 1: Conduct Grab Sampling of effluent for 24 hours at an interval of 02 (Two) hours.

Step 2: Conduct flow based composite sampling for every 24 hours. All measurements shall be reported in metric SI units. For Example, the flow shall be reported in m³/hr after conversion factor of 4.546 is applied to the readings in MGD

Step 3: Follow standard sampling procedure for

- i. flow based composite preparation.
- ii. sample collection
- iii. sample preservation
- iv. sample storage with ice
- v. samples deposition

Step 4: Send samples for testing to a EPA recognized laboratory, which has participated in AQC, within last one year and has passed the AQC criteria successfully.

Step 5: Collect sample results within maximum seven days from the date of sampling.

Step 6: Collect copy of certificate of Lab’s participation in AQC (as prescribed by CPCB) and its performance results in AQC, along-with the sample’s results each time either physically or electronically (email etc.).

- Step 7: Enter data of 12 (Twelve) Grab samples lab results of every parameter (pH, BOD, COD, TSS) into the OCEMS within seven days of sampling and maintain a record of calibration results entered in the OCEMS.
- Step 8: Ensure that there are no previous calibration results entries in the system. If there are entries, remove all entries from the system. These entries have to be removed only at the first time calibration.
- Step 9: While making calibration results entries, ensure that outliers (data points) as per given “Criteria of outliers” in this document, are not entered into the OCEMS.
- Step 10: within next seven days from the first sampling date, Repeat the exercise again following steps 1 to 7 again.
- Step 12: This time system should hold at-least total 15 valid calibration points (out of 24 data points) after calibration points entries.
- Step 13: Compare results of composite sampling with average of continuous 15 minute values received through OCEMS for both days. The composite sample results should not be an outlier.
- Step 14: If the system does not show Standard Error Mean (SEM) within prescribed criteria for any of the parameters, that particular parameter should be recalibrated, as per above procedure.
- Step 15: As COD is the principal component of measurement of these instruments, cross check results of COD of the middle dilution, as sample. If not within 5% range of prepared working solution, perform function check again for COD parameter.
- Step 16: Only (01) sample out of 24 samples (12 samples per day for two calibration days) shall be sent to SPCBs/PCCs or CPCB lab for cross check, while doing initial calibration of OCEMS in any industry. Sample should again be sent to SPCBs/PCCs or CPCB lab, when system fails in performance and is recommended for re-calibration. The SPCBs/PCCs or CPCB may accept sample only if it is preserved and transported following standard procedure. Report on basic installation and calibration of OCEMS at STPs should be sent to SPCBs/PCCs and CPCB.
- Step 17: SPCBs/PCCs or CPCB will check the results of STPs sample received with online data and may ask for repeat of basic calibration, if found to be deviating more than acceptable error in standard laboratory method.

2.3 Continuous Validation of OCEMS

The purpose of this exercise is to check the validity of earlier calibration and collection of validated calibration data points for better matrix matching and to authenticate the range of measurement coverage. Frequency of continuous validation by plant operators should be once in 15 (Fifteen) days. The data collected should also be shared through email to CPCB, SPCB/PCCs.

Function 1: OCEMS Function check as above (as per technology used) to be performed once in fifteen days or fortnightly basis.

Function 2: OCEMS Validation through routine calibration library generation, to be performed once in fifteen days or fortnightly basis.

Process of Routine Calibration Library Generation

Step 1: Pick up one sample of effluent (treated water) from the OCEMS location.

Step 2: Create data entry point in OCEMS and note down exact date and time along-with Global calibration value at this point of time.

Step 3: Follow standard sampling procedure for:

- i. sample collection
- ii. sample preservation
- iii. sample storage with ice
- iv. samples deposition

Step 4: Sample should be given for testing to a EPA recognized laboratory only, which has participated in AQC - proficiency test, within last one year and has passed the criteria successfully.

Step 4: Collect result from laboratory within maximum seven days from the date of sampling.

Step 5: Collect copy of certificate of Lab's participation in AQC (as prescribed by CPCB) and its performance results in AQC, with the sample results each time either physically or electronically (email etc.).

Step 6: Visit OCEMS and enter data point in the library- ensuring that outliers are not entered into the system. Now the system will have one entry added to library. These data points of calibration along-with global calibration value should be available at central portals of respective SPCB/PCC and CPCB.

Step 7: If data point is an outlier, data entry should not be made in OCEMS, instead repeat the process from step 1 to step 7 again on the same day by picking up the sample again.

Step 8: If two consecutive validation check samples fall out of range and remains outliers, recommend basic OCEMS calibration again through the supplier.

2.4 OCEMS Performance Check by SPCBs/PCCs

The purpose of this exercise is to check the validity of earlier calibration and collection of validated calibration data points for better matrix matching and to authenticate the range of measurement coverage. OCEMS Performance check may be conducted by authorities like SPCBs/PCCs or by any authorized agency engaged by them once in 30 days or more depending on local requirement. Performance check data may be used to verify the performance of plants w.r.t. prescribed standards.

The data collected may be shared with plant operator. Data points within the ranges, should be made part of library of the OCEMS. More number of points shall provide better results. Efforts should be made to keep information available on web portals of CPCB and SPCB transparently, updated automatically.

- Step 1: Perform function check as above, as per technology used, and routine calibration verification data Library.
- Step 2: Pick up one sample of effluent from the OCEMS location.
- Step 3: Create data entry point in OCEMS and note down exact date and time along-with Global calibration value at this point of time.
- Step 4: Follow standard sampling procedure for
 - i. sample collection
 - ii. sample preservation
 - iii. sample storage with ice
 - iv. samples deposition
- Step 5: Sample should be given for testing in own laboratory or a EPA recognized laboratory only, which has participated in AQC- Proficiency Test, within last one year and has passed the criteria successfully.
- Step 6: Collect result within maximum seven days from the date of sampling.
- Step 7: Collect copy of certificate of Lab's participation in AQC (as prescribed by CPCB) and its performance results in AQC, with the sample results each time either physically or electronically (email etc.).
- Step 8: Visit OCEMS and enter data point in the library- ensuring that outliers are not entered into the system. Now the system will have more than 16 calibration points entries.
- Step 9: If data point is an outlier data entry should not be made in OCEMS, instead repeat the process from step 1 to step 7 again on the same day by picking up the sample again.
- Step 10: If two consecutive performance check samples fall out of range and remains outliers recommend basic OCEMS calibration again through the supplier.

3.0 Other Calibration Requirements

- a. Sampling procedure
 - Precaution should be taken while taking the sample for OCEMS validation and performance verification. The water sample pick up point should be same, where OCEMS is operating.
 - Sample should be collected from the outlet of final treatment water storage tank from the instrument measuring-window location.

- Standard sampling procedure should be followed to pick up/storage/transport/preserve the samples. The samples collected should be submitted to the lab within maximum 24 hours.
- Since, exact sampling time plays significant role in the validation of data, sample pick up date and time should be endorsed in the instrument.
- Snapshot of the instrument at sample pick up time should be taken for results entry into the system later.
- Calibration status of OCEMS should be recorded by means of recording number of calibration data points available in the system. Here, data points means the results of lab against data of instrument and Global Calibration values entered in the system. Snapshot can be taken to collect these three data sets.

b. Calibration procedure:

To get better results through OCEMS, adaptation of water matrix/Pollution load is a key point which should be done through sampling as mentioned above and finally based on result multipoint Calibration should be done.

- Results of water samples given to a laboratory, during initial installation or validation or performance verification of OCEMS, for testing must be obtained within five days of sampling.
- Lab result entry must be made in the software of OCEMS within seven days against the same calibration point created at the time of sample pickup. date and time with the template snapshot taken during sampling should be used for data matching and records entry.
- Lab results can be entered in the system, only if results do not fall in criteria of outliers.
- In case results do not fall in the above criteria for two times consecutively, the system Basic installation calibration should be recommended.

4.0 Recommended Outliers:

Outliers have to be calculated on the basis of calculation of SEM (Standard error mean) in the lab results obtained during initial calibrations, continuous validation and performance verification. The SEM can be calculated through excel sheets as per formula applied: $STDEV.S(G1:Gn)/SQRT(COUNT(G1:Gn))$

The recommended Criteria of outliers derived from standard error mean limits for different parameters is as below:

- pH: SEM(Standard error mean) should not be more than 5
- BOD: SEM(Standard error mean) should not be more than 20
- COD: SEM(Standard error mean) should not be more than 10

- TSS: SEM(Standard error mean) should not be more than 10

Protocols of other technologies used in field will be developed in the next version of protocol.
