DQM Standard Operating Procedure (SOP) 9.2.1.2 (V3)

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Use of the DQM Calibration and Accuracy Checks Data Sheet

1.0 About this SOP

This guidance is a part of the Data Quality Management (DQM) System implemented by the Clean Water Team (CWT) to support collection of reliable data of known quality through the use of unique Instrument ID and Standard ID. Calibration and Accuracy Check records provide information that is essential for derivation of instrument-specific measures of accuracy, and this document shows how these records can be linked to a specific instrument. The SOP provides instructions for entering information and data into selected boxes within the formal Data Sheet provided as part of the SOP. It augments instrument-specific guidance that has been provided elsewhere (in the appropriate standard operating procedures, or SOPs) by the CWT. The current version of this SOP is focused on Calibrations and Accuracy Checks linked to measurements of temperature, dissolved oxygen, conductivity, pH, and transparency or turbidity in the field, with kits and instruments commonly used by citizen monitoring groups.

This SOP has been written for you, the Field Operator who conducts measurements, calibrations, and accuracy checks in the field. It can also be used by Field Operators and Trainers participating in periodic regional calibration sessions or in Pre-event or Post-event calibration sessions in conjunction with snapshot days and other mass-monitoring events. To use SOP-9.2.1.2(Calib) (this SOP) you will need - in addition to the testing equipment and Standard Materials - hardcopies of instrument-specific SOPs for all the kits and instruments used by your team of field operators or Event participants.

2.0 Do's and Don'ts

* Always shake your measurement device (bulb thermometer, electrode, or probe) in the tested solution or medium, and make sure you take it out of solution and put it back in several times before you read the output. Results should be read only after you have allowed the instrument reading to stabilize in the tested solution or medium.

**** Never read the output of an electrode (pH, DO, EC) or any other sensor when it is touching the bottom or sides of the container! This is true for all Standard Materials and for environmental samples as well.**

* Always record the Instrument ID with the Results of your measurements.

3.0 Generic Instructions for use of the DQM "Calibration and Accuracy Checks Sheet"

This guidance section tells you how to use the DQM Calibration and Accuracy Checks Data Sheet (included in this SOP) for keeping records of your water quality monitoring. Different instruments may require different procedures for controlling and checking the accuracy of scientific measurements. The procedures for recording and reporting are also specific to an instrument, so your instrument-specific SOP is your **essential PRIMARY guidance** that should be followed when filling out this DQM Calibration & Accuracy Checks Data Sheet. The explanation below provides some definitions to clarify what each term means, and provides generic information regarding the content of each field. Notes on areas that require special attention or instructions are also provided, as well as summaries of CWT experience where appropriate.

3.1 Relevant Definitions:

These terms are defined here because they are essential for understanding the instructions. The same definitions are also given in the Glossary at the end of the SOP (Section 4).

Instrument: a probe, electrode, reagent kit, indicator strip, or any other type of device used for field or laboratory measurements.

Accuracy Check: Comparison of the reading, or output, of a measurement device with a value believed to be the "true" value. The "true" value may be represented by any Standard Material (e.g., known natural reference conditions such as freezing point, Standard Solution, etc). An "Accuracy Check" is different from a Calibration, since it is only a comparison and does not result in an adjustment of the reading of the measurement device.

Calibration (or 'calibration adjustment'): modification of the output of an adjustable-reading instrument, to make it reflect a value that represents the "true value" (as manifested by a given Standard or by a natural value).

Standard Material: A catch-all term for Solutions (e.g., Standard Buffer), devices (e.g., Certified thermometer), or natural reference points (e.g., Water saturated with dissolved oxygen at a given temperature), that represent a value believed to be the "true" value.

Standard Solution: A solution containing a known concentration of a substance or has a known property, prepared or purchased for use in the analytical laboratory or in the field. Each bottle of these types of Standards has a **unique Standard ID,** for example "STB-EC2". Every bottle of Standard with its unique ID can be described in one or more of the following definitions:

- "**Resident Standards**" – solutions that each monitoring entity or group owns and uses routinely for calibration and/or accuracy checks.
- "**External Standards**" - solutions used in events such as Intercalibration Exercises, often brought by the QA/QC officer for comparison with the Resident Standards brought by the participating groups;
- "**Certified Standards**" include any Standard that is traceable to NIST or ASTM. Resident and External Standards can all be Certified Standards as well. A Certified Standard is considered the "ultimate authority" if valid, i.e., if the bottle was (a) used before the expiration date; (b) has been stored tightly capped; and (c) has not been exposed to extreme temperatures or sunlight.

Notes: (a) You will see the term **"Standard Reagent"** for a solution or a suspension or particles of known properties that is provided with a test kit and used as part of the measurement procedure itself (e.g., reagent for turbidity comparisons in the dual cylinder kit). This solution would not have a Standard ID but will be accounted for through the kit's unique Instrument ID.
(b) The EPA's definition for "Calibration" is, essentially, a combination of "comparison and adjustment if needed"; it is not specific enough for communication of what you did when you say "I calibrated the instrument".

Most monitoring entities or groups use their Resident Standards for routine Instrument calibration and accuracy checks, but External Standards may be also used during special events. CWT coordinators distribute Certified Standards to groups in California when possible; these bottles become the Resident Standards of the group. The DQM Calibration & Accuracy Checks Data Sheet has a placeholder for all types of Standard Materials. Note: The DQM system included an additional form called *"Data Sheet for Comparisons of Standards",* which is used by the QA/QC officer during Periodic Regional Calibration Sessions, Intercalibration exercises, or before/after mass monitoring events; that form and guidance will be provided elsewhere (look for *DQM-SOP-9.3.2.1,* to be published in 2004 or 2005) and will not be discussed in this SOP.

3.2 The DQM "Calibration and Accuracy Checks Sheet"

Before you proceed, please have the DQM "Calibration and Accuracy Checks Sheet" **hardcopy** form and the accompanying **Instructions** ("Cheat Sheet") in front of you, as printed from Section 6 of this SOP. Note that the Data Sheet has places for three vertical 'records', each dedicated to a combination of one Instrument and one Standard Material. You may need to fill in more than one 'record' for one instrument (for example, if you calibrate your pH meter against your pH 7.0 buffer and then against our pH 10.0 buffer). So use as many records and sheets as needed. Be sure to add sheet page numbers in sequence, and write the total number of pages when done.

The Project ID is very important for tracking purposes, so please make sure you add that too; the placeholder for "Event" at the top right corner can be used for any descriptor you may choose for your activity.

3.3 Cell contents

The following Table provides supplemental notes to the Instructions in the 'cheat sheet' from Section 6, for selected boxes only.

box	FIELD NAME	Supplemental notes
A	**Instrument ID**	Each measurement device must have a unique Instrument ID; it relates to the individual probe (not the multimeter box) or the batch of reagents used in a field kit. Please contact your CWT Coordinator for codes and conventions if needed.
B	**Instrument Type**	(*Adjustable or non-adjustable)* - All instruments need to be 'calibrated' to reflect the 'true' value. Many Instruments are calibrated by the manufacturer and their readings cannot be adjusted by the user. Bulb thermometers, pH strips, pH liquid indicator kits, and nutrient reagent kits with comparators all fall within this category of 'non-adjustable' instruments, as well as most thermistors. In contrast, some instruments including most electrodes have to be amenable to adjustments by the user, and they have a screw or a knob or an automatic feature that can tweak the reading. In this text (and the rest of the DQM) we use the words 'calibration' or 'calibration adjustment' for the actual action of adjusting the reading when the device is subject to Standard Material, i.e., conditions that reflect a true value.
D	**Unit**	Preferred measurement unit for dissolved oxygen is mg/l (a.k.a. ppm); percent saturation will need an additional calibration record on the Data sheet. Use the letter "u" for 'micro'; avoid using the Greek letter "miu" in electronic formats. Make sure you distinguish between uS (a.k.a. umohs/cm) and mS (a.k.a. mmhos/cm) with certainty. One milliSiemen (mS) equals 1000 microSiemen (uS).
F	**Time …**	Always write the actual time at your location; add " PDT" or "PST" if you want.
G	**Reason** for Calibration or Accuracy Check	*(examples: Routine, Pre-event, Post-event, new instrument, Intercalibration exercise)* Make sure you relate the Reason for Calibration or Accuracy Check to time-line of the event; and make clear distinction between activities before or after an event of reference (only Post-event records will be used to calculate accuracy for adjustable instruments)

box	FIELD NAME	Supplemental notes
H	**Temperature** at Calibration or Accuracy Check	This box is **not applicable** to Accuracy Checks of **temperature** measuring devices, only to the other parameters. Temperature inside the solution used for Calibration or Accuracy Check affects the outcome greatly for dissolved oxygen: it dictates the DO concentration at saturation! Therefore it must be recorded, whatever it is. Electrical Conductivity (EC) greatly depends on temperature (see discussion on specific conductance and temperature compensation in DQM-IP-3.1.3). Assuming you have automatic temperature compensation (ATC) built-in to your EC meter, here is what CWT recommends: For **routine** monitoring keep your EC Standard at as close as you can to your environmental water temperature and make sure you record it. For **mass monitoring** events such as Snapshot day, whe you do the Pre-event and Post-event calibration sessions, bring your EC standards to 25 C or talk to your coordinator about the latest directions. The effect of temperature on pH is more significant at lower temperature, so always calibrate at the temperature you anticipate in the environment and record it whatever it is. Temperature is not very relevant for turbidity per se but will affect your calibration if the Standard is cold, as a result of condensation on the vial. Bring your turbidity Standards (and samples too) to room or ambient temperature before use.
J	**Standard Material**	Each bottle of Standard solution must have a unique ID; please contact your CWT Coordinator for codes and conventions if needed. The Unique Instrument ID of the certified NIST thermometer sitting in the same bucket with your thermometer is also entered as Standard Material. Natural reference point such as saturated water and distilled-water-ice bath are OK if prepared according to protocol (see section 3.3 below).
L	**Reading in Standard Material**	The 'Reading in Standard Material' for the non-adjustable instrument is totally equivalent to the 'Reading before calibration' for an adjustable instrument: they both tell us how far we are from the True value. Check your instrument-specific SOP for the number of "significant digits", i.e., how many digits in the Result have a number that is meaningful. The box has placeholders for three repeated measurements: this is mandatory for bulb thermometers, electrodes, and probes that are simple dipped in the tested solution. Talk to your CWT Coordinator about requirements for pH strips, Winkler titration kits, etc.

box	FIELD NAME	Supplemental notes
N	**Reading after calibration**	What the instrument shows in the Standard after you have adjusted the reading should be similar to the True value (box K) except when the resolution or the instrument does not enable it (e.g., when your EC Standard true value is 1412 uS and your EC meter can only show 1410 or 1420).
P	**Comments**	This box is where you communicate anything that you deem important to share with others about process irregularities, interesting observations, problems encountered, and attempts for solving them (whether successful or not). When your data validators and data users are not there with you, they rely on your input when validating and using the data you have collected.

Notes and Comments: Instrument-specific considerations or information can be recorded as Notes (for example, related to dissolved oxygen electrode calibration) or in the Comment box. If you are performing accuracy checks for nutrient kits, and the Standard Stock solution has been diluted for the test, write the name of the dilution preparer in the comment box.

3.3 Supplemental protocols

3.3.1 Natural reference points

When you want to use "natural reference points" as your Standard Material there are several things to keep in mind.
The distilled water-ice bath (or deionized water, DI) is recommended and acceptable in many of the Forest Service-derived protocols for HoboTemps. If you choose to use it please use only DI-ice and DI-water for your bath (no salt!), and make sure that
(a) the container is insulated;
(b) the mixture always contains unmelted ice; and
(c) it is thoroughly and continuously mixed.
It makes sense to check the NIST thermometers provided by CWT in an ice bath as well.

To prepare **DO-saturated water** start with DI from a bottle held at ambient temperature for a long time, and transfer it from cup to cup with ample turbulence at least 20 times. Never use water from the tap or the fridge.

3.3.2 Electrode rinsing procedures during calibration

For better accuracy, all electrodes/probes need to be rinsed (i.e., dipped in) used Standard solution before being dipped into the new, fresh Standard. Experience shows that readings may be much closer to the true value if this is done (rather than having probes put directly into the fresh Standard after the DI rinse).

3.4 How your records will be used

The calibration and accuracy checks information you have recorded on this Data Sheet is vital for generation of data of known quality. Your records will be entered into a Project File database, and Your Trainer or Technical Leader will use them to calculate the accuracy of all the measurements you have performed with your Instruments.

4.0 Glossary for This SOP

Accuracy: The extent of agreement between an observed value (measurement result) and the accepted, or true, value of the parameter being measured.

Accuracy Check: Comparison of the reading, or output, of a measurement device with a value believed to be the "true" value. The "true" value may be represented by any Standard Material (e.g., known natural reference conditions such as freezing point, Standard Solution, etc). An "Accuracy Check" is different from a Calibration, since it is only a comparison and does not result in an adjustment of the reading of the measurement device.

Blank (Sample): A sample that contains pure water and is analyzed concomitantly with a set of environmental samples. Blanks usually include field blanks and trip blanks to assure that there was not contamination during sampling and shipping, as well as laboratory method blanks and reagent blanks, tested within the analytical procedures

Buffer (capacity): the property of a solution to resist changes in pH. A good buffer is a substance that is present in an equilibrium between two or more forms (e.g., ionized and unionized) and can compensate for changes in pH by changing its equilibrium to restore the original hydrogen ion concentration. Also see Standard pH Buffer

Calibration (or Calibration Adjustment): The action of adjusting the readings of an instrument to have them match a "true" value as represented by known natural conditions (e.g., freezing point) or by a Standard Solution (e.g., Standard pH buffer).

Certified Standard: any Standard that is traceable to NIST or ASTM (also see "Standard Solution" below). Resident, Calibrator, and External Standards can all be Certified Standards as well. A Certified Standard is considered the "ultimate authority" if valid, i.e., if the bottle was (a) used before the expiration date; (b) has been stored tightly capped; and (c) has not been exposed to extreme temperatures.

Characteristic: see Parameter.

Data Quality Objectives (DQOs): Statements about the level of uncertainty in data that a decision-maker is willing to accept to support a particular decision. DQOs include measurement quality objectives (precision, accuracy, detection limit, and resolution) as well as measures of completeness, representativeness, and comparability.

Data Validation: The process to assure that the test or analysis used to generate the data was valid and that the data meets requirements and quality objectives.

Duplicate Samples: two samples taken at the same time from the same point that are carried through all assessment and analytical procedures in an identical manner.

Instrument: a probe, electrode, reagent kit, indicator strip, or any other type of device used for field or laboratory measurements.

Parameter: A property or substance to be measured within a medium. Parameters include properties such acidity (pH) or electrical conductivity, particulates such as suspended solids or bacteria, and analytes such as ammonia or heavy metals.

Precision: A measure of how close repeated measurements are to each other.

Resolution: The smallest increment that can be discerned on the scale of a measuring device, or the capability of a method to discriminate between measurement responses.

Significant digits - digits in a numerical Result that have a number that is meaningful. In most cases three significant digits are fine, e.g., 10.4 mg/l DO (all three digits are significant) or 1560 uS (the first three are significant, the last one provides the order of magnitude but the difference between zero and, say, 2, is not significant).

Standard Operating Procedure (SOP): A written document providing step-by step instructions for performing a procedure (sampling, measurement, or other).

Standard pH Buffer: A well-buffered solution used for calibration or for accuracy checks of pH measuring devices (also see "Standard Solution" below).

Standard Reagent: A solution or a suspension or particles or known properties, such as concentration or turbidity, that is provided with a test kit and used as part of the measurement procedure (e.g., reagent for turbidity comparisons in the dual cylinder kit).

Standard Reference Materials (SRM): An SRM is a certified material or substance with an established, known and accepted value for the analyte or property of interest. Many SRMs are produced by the U. S. National Institute of Standards and Technology (NIST) and characterized for absolute content independent of any analytical method. They are often used as an external "Accuracy Check" to determine if there is any bias due to the materials used for calibration, or to validate the internal Laboratory Control Standards. Some people use the term "Certified Reference Material" for the same entity.

Standard Solution: A solution containing a known concentration of a substance, prepared or purchased for use in the analytical laboratory or in the field. It is used in calibrations and in quality control checks on procedures and instruments. This definition includes Standards used for accuracy checks of all types of measurement devices and for calibration adjustments of adjustable pH and conductivity meters (etc.). Solutions can be the "Resident Standards" that each monitoring entity uses, as well as SRMs and "External Standards" such as those used in regional Intercalibration Exercises. Each bottle of a Standard Solution may be identified specifically via a unique ID.

5.0 Sources and Resources

This SOP is an integral part of the Data Quality Management (DQM) System implemented by the Clean Water Team, the Citizen Monitoring Program of the California State Water Resources Control Board.

For an electronic copy, to find many more CWT guidance documents, or to find the contact information for your Regional CWT Coordinator, visit our website at www.swrcb.ca.gov/nps/volunteer.html

If you wish to cite this SOP in other texts you can use "CWT 2004" and reference it as follows:
"Clean Water Team (CWT) 2004. Use of the DQM Calibration and Accuracy Checks Data Sheet, DQM SOP-9.2.1.2(V3) in: The Clean Water Team Guidance Compendium for Watershed Monitoring and Assessment, Version 2.0. Division of Water Quality, California State Water Resources Control Board (SWRCB), Sacramento, CA."

Note: The "DQM Calibration and Accuracy Checks Data Sheet" has been revised and refined by the CWT three times since 2000, and the present guidance supports the third iteration of the Data Sheet (even though it is included in V2 of the entire Compendium).

6.0 The Form and Instructions

(see next 2 pages)

DQM Calibration and Accuracy Checks Data Sheet V3 **Event** _____

Project ID _____ Page _____ of _____

		Record 1	*Record 2*	*Record 3*
A	**Instrument ID**			
B	**Instrument Type** *(adjustable or non-adjustable)*			
C	**Characteristic (Parameter)**			
D	**Unit**			
E	**Date** of Calibration or Accuracy Check			
F	**Time** of Calibration or Accuracy Check			
G	**Reason** for Calibration or Accuracy Check (*Pre-event, Post-event, Routine, new instrument*)			
H	**Temperature (C)** at Calibration or Accuracy Check (not applicable for temperature checks)			
I	**Thermometer ID** (not applicable for temperature checks)			
J	**Standard Material** (enter *Standard ID, or NIST thermometer ID, or 'saturated water', or 'distilled water ice-bath', etc.)*			
K	**"True" value of Standard Material** [see on the label] or **natural point**			
L	**Reading in Standard Material** (non-adjustable instruments) or **Reading in Standard Material before Calibration** (adjustable instruments)	**first** **.** **second** **.** **third**	**first** **.** **second** **.** **third**	**first** **.** **second** **.** **third**
M	**Action taken** *(cal, none, or nap)* (adjustable instruments only)			
N	**Reading after calibration** (adjustable instruments only)			
O	**Cal/AccurCheck Operator** *(your name)*			
P	**Comments**			

Note: If you are calibrating an oxygen electrode, write "humid air" or "saturated water" in the "Standard Material" box above.

If you calibrate in air and can measure the absolute barometric pressure, write the value and unit here _____; otherwise please indicate your elevation above sea level _____.

Instructions for the DQM Calibration and Accuracy Checks Data Sheet **'Cheat Sheet'**

	Field Name	Content	Examples
	Project ID	Write the ID of the Project	VAR03; SPC02; SNP03
A	**Instrument ID**	Write the instrument ID as it is written on the instrument	DOW-STB01; PHP-2BR6
B	**Instrument Type**	Write whether the instrumnet is adjustable (e.g., conductivity, pH, or dissolved oxygen electrodes; turbidimeter) or non-adjsutable (e.g., bulb thermometer, pH strip, Windkler kit)	adjustable
C	**Characteristic (Parameter)**	Write the name of the characteristic (parameter) you are measuring or calibrating for	Dissolved oxygen; pH
D	**Unit**	Write the measurement unit	Degree C, pH, mg/l; uS
E	**Date** of Calibration or Accuracy Check	Write the date (mm/dd/yy)	04/25/01; 03/02/98
F	**Time** of Calibration or Accuracy Check	Write the time of day, to the nearest minute, preferably using a 24 hour clock (but AM PM is OK if you add that).	14:36, 4:30 PM
G	**Reason** for Calibration or Accuracy Check (*Pre-event, Post-event, Routine, intercalibration*)	Write the reason for doing the calibration or accuracy check, and whether it is before the event of reference or after the event.	Post-event
H	**Temperature (C)** at Calibration or Accuracy Check (not for temperature checks)	Write the temperature as measured at the same time inside the solution used for Calibration or Accuracy Check (this is essential for dissolved oxygen, conductivity, and pH, optional for turbidity). **Write "nap" (not applicable) for temperature accuracy checks.**	25 C
I	**Thermometer ID** (not for temperature checks)	Write the ID of the thermometer you are using to check the temperature of the Standard Material during calibration or accuracy check. **Write "nap" (not applicable) for temperature accuracy checks.**	TR-RK06; TTP-STB02
J	**Standard Material** (enter *Standard ID, or NIST thermometer ID, or 'saturated water', or 'distilled water ice-bath', etc.)*	Write the ID of the Standard Solution or the NIST thermometer you are checking against, or the material that represents your natural reference point.	STB-PH23a; 3NET-EC4; Ice-water bath; Saturated water, TR-STB43
K	**"True" value of Standard Material** *[see on the label]* or **natural point**	Write the value specified for the Standard, or the value expected at the natural reference point used as the Standard Material	7.00; 1413 @ 25 C; 8.7 mg/l; 19 C
L	**Reading in Standard Material** (non-adjustable instruments) or **Reading in Standard Material before Calibration** (adjustable instruments)	Wait for stable reading and then write the first result that the instrument shows in the Standard Material; take it out and back in for the second reading, and again for the third (check your instrument-specific SOP for the number of significant digits to be recorded in the Results).	9.8; 1390; 7.08
M	**Action taken** (adjustable instruments only)	Write "none" if you have not adjusted the reading and skip the next field; Write "manual cal" if you have adjusted the reading manually, or "auto cal" if the instrument has an automatic calibration feature; write "nap" for non-adjsutable instruments.	None; auto cal
N	**Reading after calibration** (adjustable instruments only)	Write the result that the instrument shows in the Standard Material, after you have adjusted the reading (it should be similar to the True value, above)	1410; 447
O	**Cal/AccurCheck Operator** *(your name)*	Write your first initial and full last name	D. Johnson
P	**Comments**	Write anything that you deem important to share with your leader, QA/QC officer, or data user	The Standard was not at 25 C