SOCIB Balearic Islands Coastal Observing and Forecasting System

Analytical determination of Salinity with laboratory Portasal 8410A

SOCIB-Systems Operation and Support Division

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1.0.0	2018-05-21	First version document	C. Muñoz	J. Allen/E.Alou
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1. Introduction

Salinity is a fundamental EOV (Essential Ocean Variable) for water mass property determination and characterization. SOCIB instruments and platforms measure practical salinity electronically according to international standards. *In situ* water bottle sample salinity determination is essential for validation of these multi-platform observations, for the validation process and for the determination and correction of long-term sensor drifts.

The aim of this document is to describe the standardized procedure used in SOCIB to conduct the analytical determination of seawater salinity from water bottle samples operating a Guildline Portasal 8410 salinometer.

The analytical results obtained from the *in situ* water samples will be used for a delayed mode field calibration of the ship-based lowered and underway CTD sensors. This follows the Data Management Plan, which references, with links, all the Standard Operating Procedures, including the SOP Salinity Correction Data Post Processing.

2. Related documents

User's Manual portasal 8410A

Portasal 8410A Portasal 8410A Specification sheet

SPEC_DCF_project-management-naming-convention

SPEC_DCF_SOCIB_raw-data-file-naming-convention

RV water sampling protocol (see this document for details on sample labeling convention)

3. Required material

- Portasal 8410A.
- Portasal spares kit.
- Laptop with the portasal software (Salinometer Data Logger).
- Standard seawater P-Series IAPSO (~ 35.000 salinity, OSIL)
- Standard seawater 38H-Series IAPSO (OSIL).
- Distilled water (approx. 20 L).
- 0.5 L Triton cleaner dilution (0.01% 1 ml Triton/1 L distilled water).
- CTD-cast operator logbook (in case the samples were obtained from a field cruise) or

any other field logbook which includes the required sampling details of the seawater samples.
- Field salinity spreadsheets (used during the cruise).
- Laboratory salinity analysis logbook (to be filled during the analyses in the lab).
- Sea water samples for salinity analysis.
- Thermometer (to track the laboratory temperature).
- Dehumidifier.
- Plain screwdriver.
- Paper towel roll.

4. Preparation steps (to be done the day before the analyses)

This stage of the procedure it's done 24 hours before the seawater sample analysis.

4.1. Temperature controlled room

1. Clean the working area.
2. Set up the laboratory space and bring the salinity standards needed and the seawater samples to be analyzed.
3. Switch on thermostat in order to acclimate the room around 21 ºC (1-2 degrees below the portasal water bath temperature) .
4. Switch on dehumidifier in order to acclimate the room at around Hr= 50-60 %. Remember to empty the dehumidifier from time to time.
5. Leave the temperature sensor tracking overnight and check on the day that the analyses are planned (the temperature should be fairly stable).

Figure 1. Laboratory set up for *In situ* water bottle sample salinity determination (temperature controlled room).

4.2. Portasal assembly and preparation

1. Open the portasal salinometer back lead with the screwdriver.
2. Connect the power supply and all connectors to the laptop (for software communication).
3. Open the portasal salinometer front lead and place the distilled water tank on top of the portasal (as shown in Figure 2).

Figure 2. Portasal assembly ready for the preparation of the analytical determination routine, the image shows filling up the system with distilled water.

4. Connect the peristaltic pump power supply cable.
5. Connect the plastic tubing to the portasal tank drain/fill and overflow spigots (see Figure 3).

Figure 3. Overflow tubing connected to Tank drain/full and overflow spigots

6. With the portasal switched off, fill the portasal tank with distilled water through the tank drain/fill (this is done by gravity and it is the reason for placing the distilled water bottle on top). This should take around 10 to 15 minutes until you observe water through the overflow tube (see Figure 2).
7. Once the portasal is full with distilled water, disconnect from spigot first the tank overflow tube to avoid any bubble formation and second disconnect the tank drain/fill (by pushing the metallic button).
8. Turn on the portasal.
9. Press key 1 T set (see figure 4).

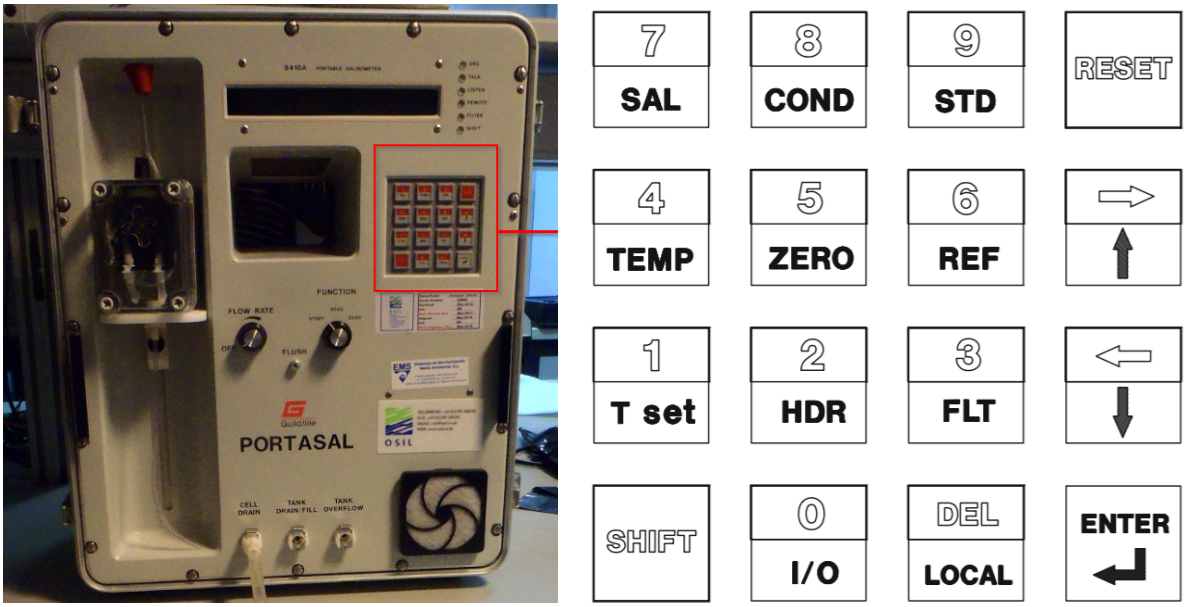


Figure 4. Portasal instrument front characteristics and magnified image with detailed numeric keys and arrows in the keypad.

10. Set up the portasal water bath temperature at 23ºC (1 to 2 ºC above the highest registered room temperature at most). Select the desired temperature using the key arrows (↑↓ Figure 4).
11. Press ENTER in the key pad panel (see Figure 4).
12. Connect the plastic tubing to the cell drain spigot in order to allow the water to exit the system.
13. Fill the portasal circuit with the Triton dilution and flush. Repeat this process 10 times.
14. Fill the portasal circuit with distilled water and flush. Repeat this process until 1 L of distilled water is used.
15. Leave the portasal circuit filled with distilled water.
16. Leave the portasal turned on (to temperate the water bath overnight).

5. Analytical determination

1. Remove any air bubbles that could be present in the water bath by refilling the system with distilled water. Follow steps 5 and 6 in section 4.2. in order to do this (Portasal assembly and preparation).

5.1 Prepare file

1. Turn on the laptop.
2. Switch portasal Function to Standby position (STDBY, as shown in Figure 5).

Figure 5. Detail of the Function switch to selectin the various positions in the instrument

3. Open the salinometer software named “Salinometer Data Logger” from the laptop.
4. Open a new file.
5. The software will show the message as in Figure 6 to assure that the communication settings are correct.

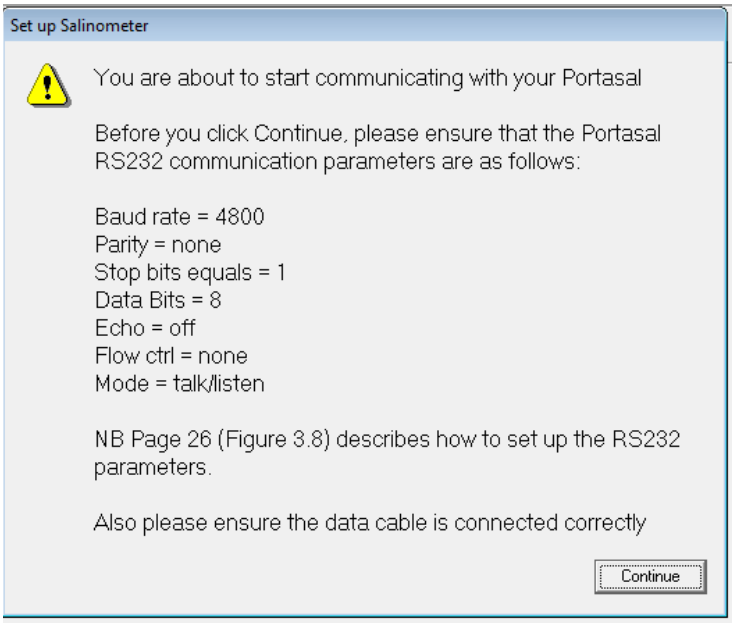


Figure 6. Message displayed in the laptop to check the configuration settings before starting the analyses

6. Check and follow the instrument configuration for the communication of the instrument (as shown in Table 1). If some parameters are not as in Table 1, please go to section 3.6.1 in the manual and follow guidelines.

KEY PRESS	DISPLAY
I/O SETUP	RS-232C BAUD RATE
ENTER	BAUD RATE 4800
ENTER	RS-232C BAUD RATE
↓	RS-232C PARITY
ENTER	PARITY NONE
ENTER	RS-232C PARITY
↓	RS-232C STOP BITS
ENTER	STOP BITS 1
ENTER	RS-232C STOP BITS
↓	RS-232C DATA BITS
ENTER	DATA BITS 8
ENTER	RS-232C DATA BITS

↓	RS-232C ECHO
ENTER	ECHO OFF
ENTER	RS-232C ECHO
↓	RS-232C MODE
ENTER	MODE TALK/LISTEN
ENTER	RS-232C MODE
↓	RS-232C EXIT
ENTER	SETUP RS-232C
↑	SETUP EXIT
ENTER	

Table 1. Steps to check the portasal configuration for the communication of the instrument (instrument/software).

7. If the configuration is correct then select continue (Figure 6).

8. The software "Salinometer Data Logger" will show a window for a new sample run (see Figure 7). The analyst will need to fill the required information: Run details such as Run ID, File Name, where to save the generated files (by selecting the … button), General Details such as ship's name, cruise number, analyst etc. Figure 7 (right panel), shows an example used for an external project, in the case of the Canales monitoring line, the ship's name is R.V.SOCIB and Cruise number SOCIB-CanalesSeasonYY.

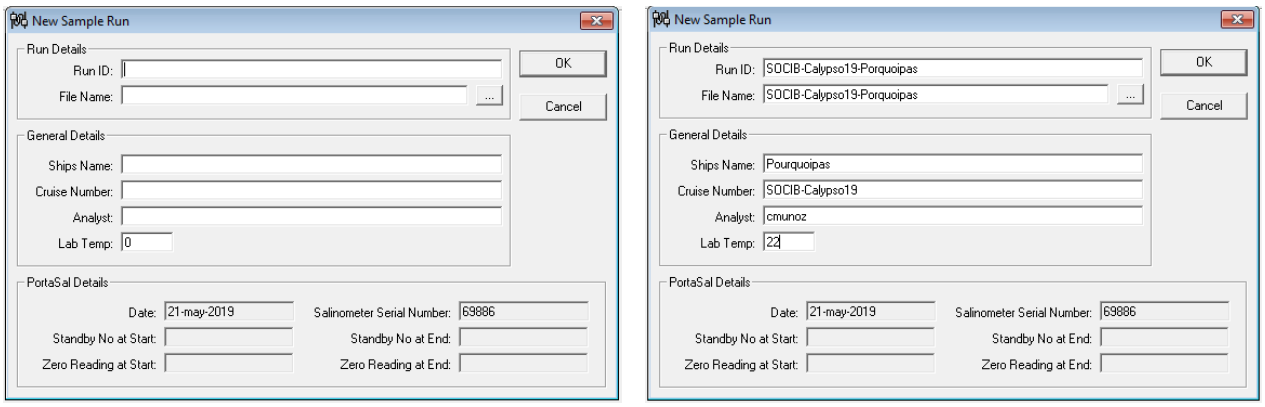


Figure 7. Windows displayed in the software Salinometer Data Logger, left panel will need to be filled as in the right panel (the example presented here belongs to an external project).

9. A new folder should be created for each analyses and all *in situ* water bottle sample batches will be saved in one folder only (i.e. one folder is generated each field campaign SOCIB-NameYY).
10. After all the fields are filled, select OK.
11. Check the zero for the conductivity reading in the portasal. Select Function Switch to the zero position (ZERO, Figure 5), in this moment the instrument is reading the conductivity of the distilled water running in the system, the value should be 0.00000 ± 0.0001. If this is not the value displayed, check for any potential contamination. Once this is verified and there is no source of contamination, then a zero calibration should be performed. see section 3.5.3 in the manual and follow guidelines.
12. Wait till the zero is measured (the laptop screen will show this).
13. Switch to Function "standby" (Figure 5).

Note. In the following sections, the peristaltic pump should be kept at its optimal flow rate (level 3 in our case) and the portasal should always be kept at the maximum flow rate (see Figure 8).

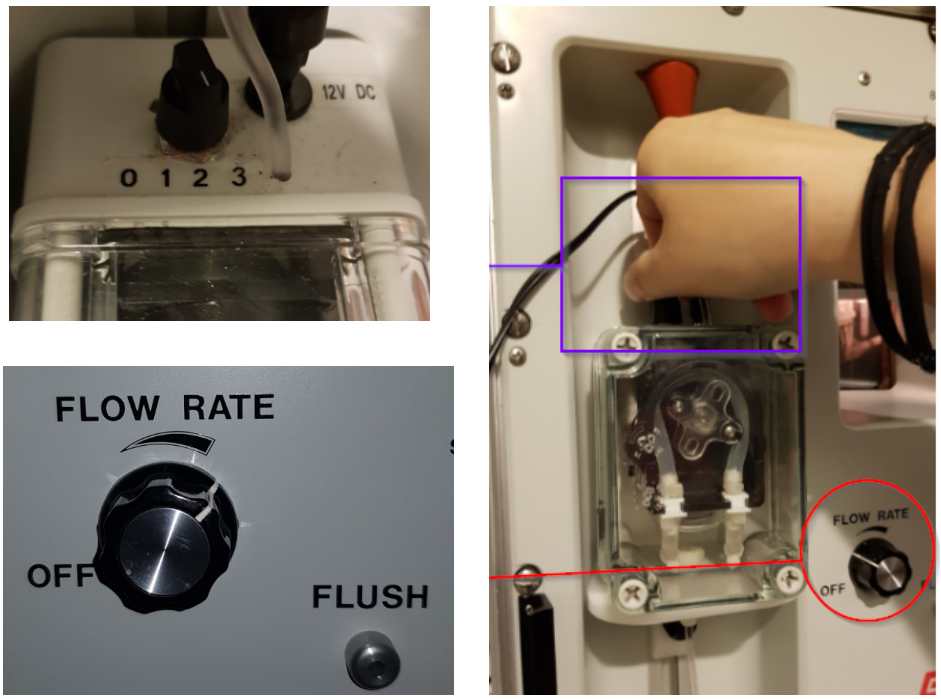


Figure 8. Valve to regulate the peristaltic pump flow rate (top left panel) and its location in the instrument (top right panel, in purple). Valve to regulate the instrument flow rate and hole flush (bottom left panel), its location in the Portasal is shown in the bottom right panel (marked in red).

5.2. Standardization

1. Homogenize the sample by gently rocking the sample by hand for a few minutes, then keep the sample settling (with no movement) for 4 to 5 min before reading. The later step helps to remove any bubbles present before performing the reading.
2. Make sure that the room is at the right temperature (in our case at 21ºC).
3. Change the Function Switch to “standby” (STDBY, Figure 5).
4. Fill the circuit with the P-Series IAPSO standard seawater (OSIL, see Figure 9) and flush placing fingertip over the flush hole (Figure 8). Repeat the process 3 times.

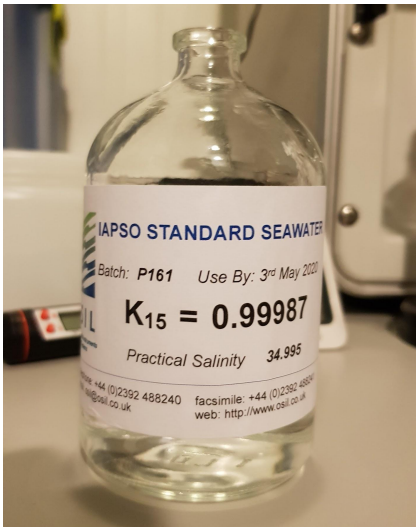


Figure 9. P-Series IAPSO standard seawater bottle with the K_{15} labeled value.

5. Select key 9 STD by pressing ENTER (see Figure 4). Introduce the batch code and the K_{15} value in the portable salinometer. The portasal will ask for this information at this step after pressing key number 9.
6. Fill the circuit with the P-Series IAPSO standard seawater (OSIL) to proceed to the reading.
7. Switch Function to “Read” (Figure 5).
8. Press ENTER and wait for the stabilization of the measurement (remember, this value is not a conductivity value but a reference ratio).
9. Once the reading is stable, press ENTER. Now this reading its the conductivity value and it should be as close as possible to the K_{15} standard bottle labeled value provided by OSIL (see example in figures 9 and 10 where K_{15} = 0.99987).

Note. If the obtained reading value does not correspond to the expected value, please repeat from steps 5 to 9 in this section until the value is correct.

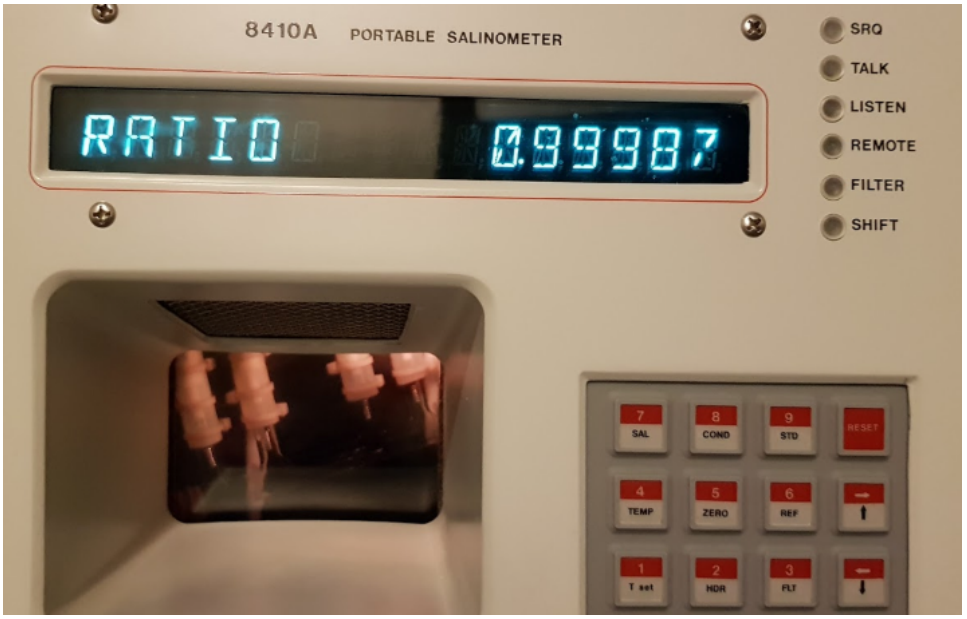


Figure 10. Instrument display with the K_{15} standard conductivity value.

The following steps define how to save this calibration in the computer software Salinometer Data Logger.

10. Select the erlenmeyer icon (see icon circled in red in figure 11) in the software "Salinometer Data Logger" (see icon circled in red in figure 11) and a new window will open to fill the calibrate salinometer ratio reading requested information (see Figure 12).

Figure. 11. Window to save the standardization in the portasal software "Salinometer Data Logger".

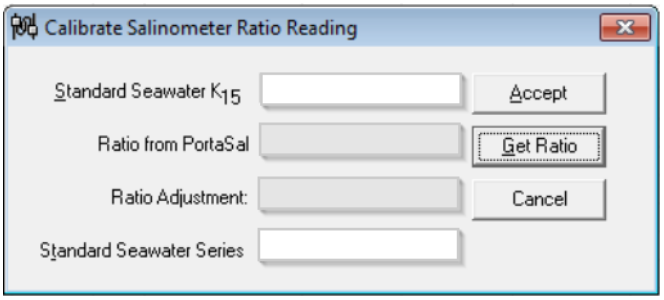


Figure. 12. Open window after selecting the erlenmeyer icon to fill with the requested information (i.e. K_{15} value and Standard Seawater series label) for the salinometer calibration ratio reading.

11. Run the calibration standard with the Salinometer Data Logger software by introducing the Standard Seawater K_{15} value and the Standard Seawater P-Series Label (P162 in this example) and select Get Ratio.

12. After the previous step the software displays the obtained P-Series IAPSO Standard Seawater reading from the portasal instrument (see Figure 13).

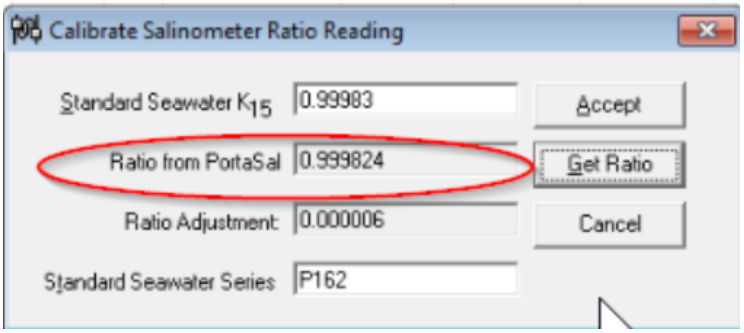


Figure 13. Obtained reading displayed in the portasal software Salinometer Data Logger.

13. If the value is correct then select Accept in the window. If the displayed value is not correct then repeat the standardization (steps 5 to 9 and 11 and 12 from this section).

Now that the calibration is accepted in the software, the following section is to reassure the correct calibration by obtaining the expected theoretical P-Series IAPSO standard seawater bottle practical salinity value.

14. Switch portasal Function to "Read" (the instrument takes around 10 s for stabilization and takes 10 small measurements).

15. Press button 7 SAL for the salinity reading display (figure 4).
16. Flush and fill the circuit and repeat 2 more times to obtain 3 readings.

Note. In case the obtained value is not the expected for the P-Series IAPSO standard seawater bottle, please repeat steps from 4 to 17 (in this section).

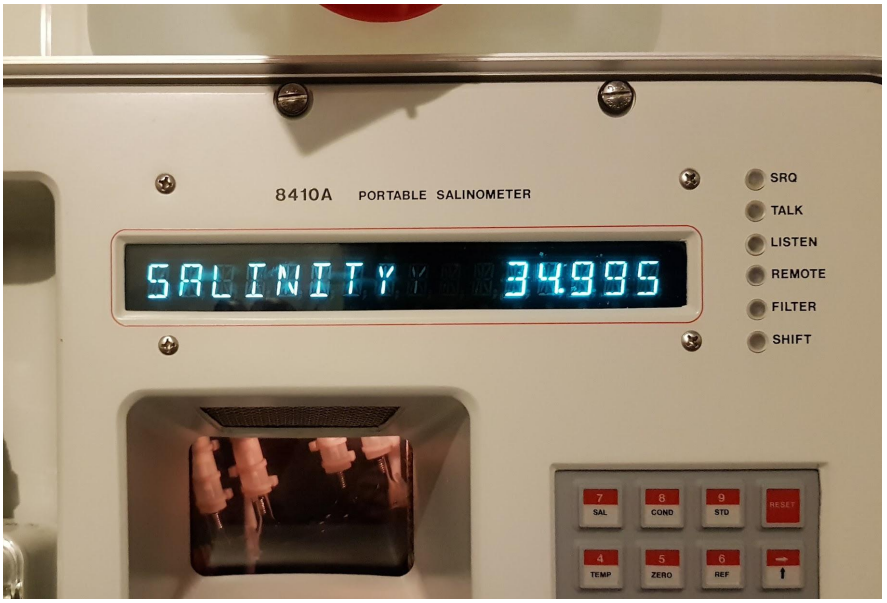


Figure 14. Display of the obtained value for the P-Series IAPSO standard seawater.

5.3. Linearity check

In order to perform a check for any potential malfunction of the instrument and verify the performance of the portasal in low and high ranges a linearity check is performed. This test should follow the first standardization of the day of analyses and consists in reading the 38H-Series IAPSO Standard Seawater (OSIL), right after the first standardization of the day. If more than one day is needed to complete the analyses then a new check should be done (one check every analytical working day). The following steps summarize the procedure:

1. Homogenize the 38H-Series IAPSO standard seawater (OSIL) sample by gently rocking the sample by hand for a few minutes, then keep the sample settling (with no movement) for 4 to 5 min before reading. The later step helps to remove any bubbles present before performing the reading.

2. Fill the circuit with the 38H-Series IAPSO standard seawater (OSIL) and flush. Repeat the process 3 times.
3. Fill circuit with the 38H-Series IAPSO standard seawater (OSIL) for the reading.
4. Switch portasal Function to "Read" (Figure 5, the software takes around 10 s for stabilization and takes 10 small measurements).

5. Press key 7 SAL (Figure 4, for the salinity reading display).
6. Flush the circuit.
7. Repeat 2 more times to obtain 3 readings.

Note. In case the check gives unclear or bad results the analyst should contact the portasal service (OSIL).

5.4. Preparation for running the samples

1. Homogenize the sample by gently rocking the sample by hand for a few minutes, then keep the sample settling (with no movement) for 4 to 5 min before reading. The later step helps to remove any bubbles present before performing the reading.
2. Before running any the sample (peristaltic pump on), make sure the previous sample exits the circuit to avoid any contamination and wipe the suction tube (see Figure 15).
3. Open the bottle sample and place in holder, inserting pick-up tube into the bottle as shown in Figure 15 . The pickup tube mustn't touch any part of the bottle while analyzing the sample.
4. Fill the circuit with the first seawater sample and flush. Repeat the process 3 times.

Figure 15. Pick up tube whipping between samples to avoid contamination (left panel), Sample ready to be runned (right panel).

5. Make sure the portasal switch stays in Function "standby".

6. Fill up the cell with the current sample, one should always check for the presence of bubbles in the cell and if bubbles are present then flush the cell and refill again until no bubbles are present in the system (see Figure 16).

Note. Tiny little bubbles that repeatedly appear may cause significant effects in the result (the values should be considered as incorrect).

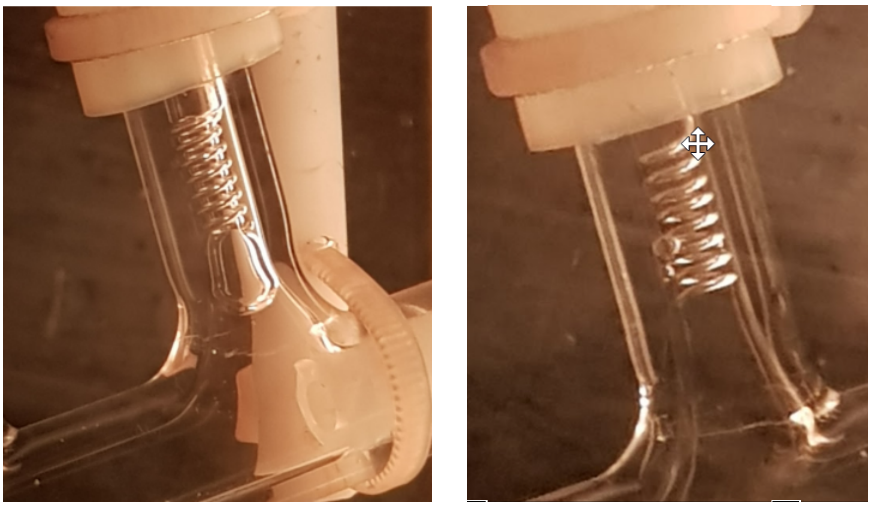


Figure 16. This figure shows an example of bubbles present in the cell.

Note: in case some bubbles are present in the last sample run, the file reading must be removed from the Salinometer Data Logger software since it is considered a bad value.

7. Turn off the peristaltic pump.
8. Switch to function “read” (Figure 5, the software takes around 10 s for stabilization and takes 10 small measurements.
9. Select key 7 SAL (Figure 4) to obtain the salinity value in the display and write the obtained value in the laboratory logbook (this step is important as serves as a backup for the results).
10. Flush the circuit.
11. Repeat steps 5 to 10, two more times to obtain 3 readings. After the 3 obtained readings for each analysed sample, the Salinometer Data Logger software will show the following message “Do you want another Reading from this bottle?” (see Figure 17), if you press **NO,** the software will save the current readings. If you press **YES** the current readings will not be saved and the software will perform a new reading with the same sample again.

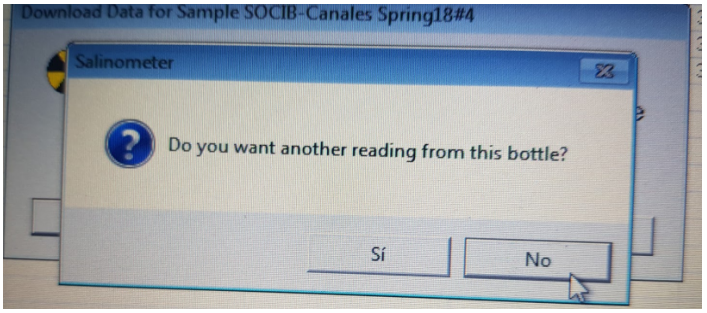
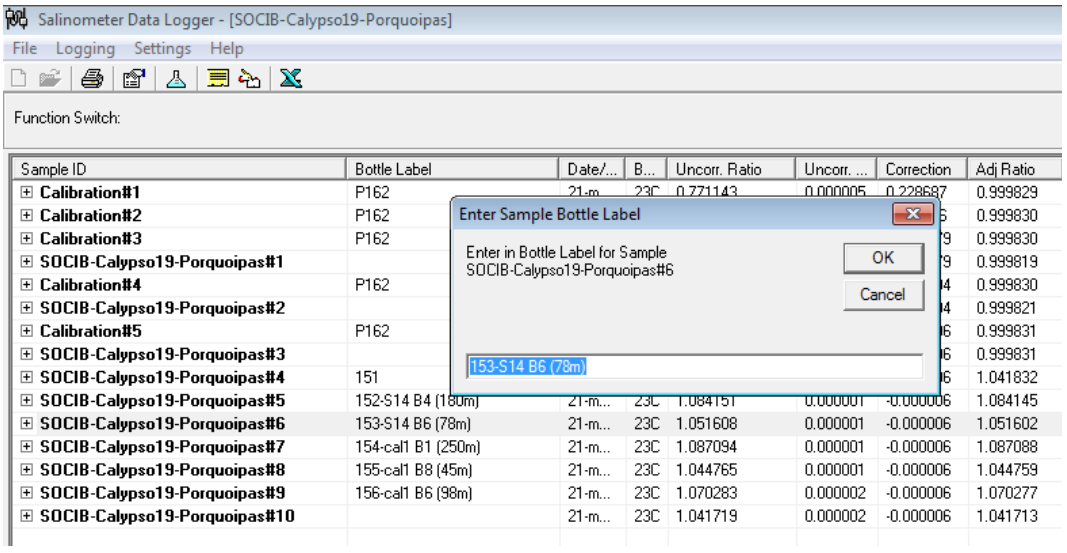


Figure 17. Window opened before saving the analytical results.

12. After the last reading a new line will appear with the stored information, the bottle label for each water sample will need to be introduced in the software (see Figure 18), to do this, use the right button in the mouse and a new window will appear to introduce the sample bottle label, comments etc. This information needs to be properly completed and should include the salinity sample bottle number, station name, niskin bottle number and depth (e.g. 52-RDM_01 B2(55m)).

Note. The station labelling convention for the SOCIB Endurance canales line (ENL) at this step of the analytical determination is: for the Mallorca channel samples RDM_xx and for the Ibiza channel S2_xx for other projects please see the specific conventions.



Sample ID	Bottle Label	Date/...	B...	Uncorr. Ratio	Uncorr. ...	Correction	Adj Ratio
Calibration#1	P162	21-m...	23C	0.771143	0.000005	0.228687	0.999829
Calibration#2	P162						0.999830
Calibration#3	P162						0.999830
SOCIB-Calypso19-Porquoipas#1							0.999819
Calibration#4	P162						0.999830
SOCIB-Calypso19-Porquoipas#2							0.999821
Calibration#5	P162						0.999831
SOCIB-Calypso19-Porquoipas#3							0.999831
SOCIB-Calypso19-Porquoipas#4	151						1.041832
SOCIB-Calypso19-Porquoipas#5	152-S14 B4 (180m)	21-m...	23C	1.084151	0.000001	-0.000006	1.084145
SOCIB-Calypso19-Porquoipas#6	153-S14 B6 (78m)	21-m...	23C	1.051608	0.000001	-0.000006	1.051602
SOCIB-Calypso19-Porquoipas#7	154-cal1 B1 (250m)	21-m...	23C	1.087094	0.000001	-0.000006	1.087088
SOCIB-Calypso19-Porquoipas#8	155-cal1 B8 (45m)	21-m...	23C	1.044765	0.000001	-0.000006	1.044759
SOCIB-Calypso19-Porquoipas#9	156-cal1 B6 (98m)	21-m...	23C	1.070283	0.000002	-0.000006	1.070277
SOCIB-Calypso19-Porquoipas#10		21-m...	23C	1.041719	0.000002	-0.000006	1.041713

Figure 18 Example of a Salinometer Data Logger generated file after a salinity analysis.

13. Repeat all the steps from 1 to 12 for each water sample.

Note. In case the analysis should be interrupted for more than 5 minutes, change the function switch to “standby” (STDBY), clean the circuit with distilled water and leave it filled

with distilled water.

5.5. Finalizing the analyses

1. To check for potential drifts in the instrument performance, run the P-Series IAPSO standard seawater in the same way as if it was a sample and compare the obtained readings in the standardization steps of the begin of the analysis. The measurements should reflect the standard expected values. In case of any contradictory results these should be notified. These could be related to standard problems and therefore the standard should be doubled checked (use a new standard bottle) or the analyses will need to considered bad. Before considering this, check for any potential source of contamination such as not wiping between samples or leaving the standard bottle open (since can change its properties).
2. Close the file in the Salinometer Data Logger software.
3. Switch function to “zero” (Figure 5)
4. The final step should be cleaning the circuit with distilled water and repeat the process at least 3 times (Figure 19).

Figure 19. Distilled water placed in holder with the inserted pick-up tube to clean and fill the circuit.

5. After the third flush, fill the circuit with distilled water and leave it.
6. If more batches of seawater samples need to be analyzed the next day then leave it as shown and next day start with the standardization (section 4.4).
7. If all samples of a program are analysed then switch off portasal and follow to the next final step.
8. Empty the portasal water tank but leave distilled water in the cell until next time.

6. Data archival and storage

Three data files are generated after each analytical determination with the following file extensions:

1. Filename.**raw**. This is a binary file that includes all the information.
2. Filename.**hdr**. File containing all the analyses metadata.
3. Filename.**dat**. This file contains the obtained analytical results.

The obtained data files (all 3 formats, .hdr, .dat, .raw) need to be exported to an excel format (.**xlsx**). This makes it easier to work with the final data. The last step of the analysis is actually the production of this file nameArchive**.xlsx** (see example). In order to do this the software Salinometer Data Logger needs to be open.

The file ID is **YYYY-MM_identification** where YYYY-MM is the starting analyzing date and the identification is generally the label for the field cruise, field work or sampling.

Additionally, and as supporting documents both the field and laboratory logbooks are scanned and uploaded together with the data final file in a pdf format.
The ID and file extension of the two files is **YYYY-MM_identificacion.pdf** where for the field logbook the date is the initial sampling date and for the lab logbook the analyses starting date-

Once the analyses are finished and the documents scanned the next step is to perform a backup of the obtained files in the MiniNAS04 (IMEDEA) with the following path \\mininas04\DataUser\DATOS\SOCIB\Análisis de laboratorio\Salinómetro.

The file will be named with the previously detailed convention ID: YYYY-MM_identification.

The 6 final files will have to be uploaded to the SOCIB google drive with the following paths depending on the platform the sampling is related to:

1. **Field oceanographic cruise**s
> Campañas > Research Vessel > CruiseName > Analysis > Salinity

2. Analytical determination on water samples obtained in the **fixed stations**
Análisis de laboratorio > Salinidades>

3. **Test samples or experiments**
Análisis de laboratorio > Salinidades>

In a final step and to assure that the data are properly stored in SOCIB, the obtained final **.xlsx** file is uploaded to the data file system following the path /socib/raw/DT/vessel/LABORATORY/SAL/ and is archived in accordance with the naming conventions SPEC_DCF_SOCIB_raw-data-file-naming-convention.

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7. Final Recommendations

The stated measurement range of the Portasal™ (model 8410A, Guildline) is from 0.004 mS/cm to 76 mS/cm with a resolution of 0.0003 mS/cm and the accuracy of the measurement is better than 0.003 equivalent Practical Salinity Units (see the User's Manual portasal 8410A).
In order to ensure the quality of the analyses some quality assurance procedures need to be considered. For this reason, the present protocol includes a zero check, a zero calibration if needed and a linearity check.
Additionally and in order to validate the method, participation in intercomparison exercises is encouraged. SOCIB will perform comparison tests with other institutions when there is opportunity and will participate in international intercomparison tests (for example in the frame of the JERICO-NEXT project, in which SOCIB scored very highly).

8. References

Guildline Instruments. (2007). Technical Manual for Model 8410A Portasal™

UNESCO. (1978). Technical Papers in Marine Science, 28, 35pp.

9. Supplementary information

The following section defines the configuration settings used by the computer software (Salinometer Data Logger). The defined general settings should be as follows (see Figure 20):

Number of readings per bottle: 3
Number of values per Reading: 10
Maximum Standard Deviation: 0.0003
Temperature Units: in celsius
Logging Settings take reading: 10 s

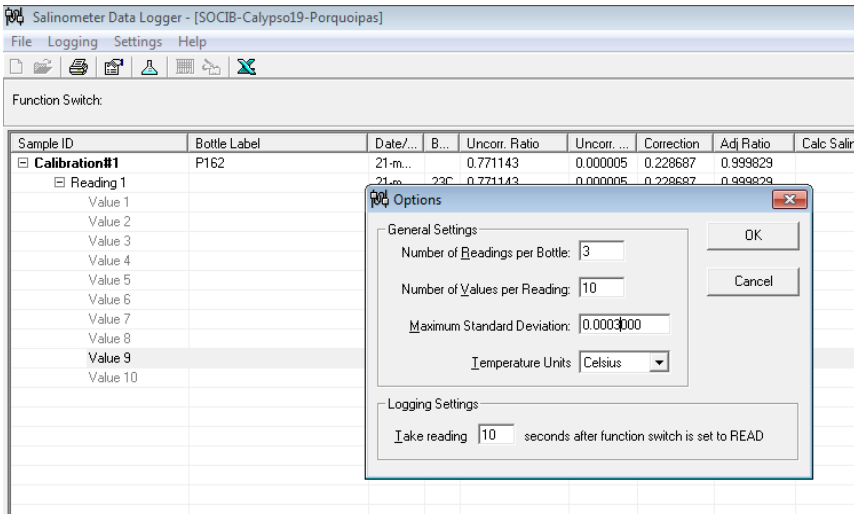

Figure 20. Defined general settings as they should be included in the computer software.