MARINE SCOTLAND - SCIENCE LABORATORY MANUAL	**OS SOP SAL** **Issue No**	Error! AutoText entry not defined.
Standard Operating Procedure: Salinities	**Issued By** **Date of this Issue:**	**1.00** **Matt Geldart** **16/02/2009**

1. **Introduction and Scope**:

This Procedure document details all procedures regarding salinities within Marine Scotland – Science:

2. Principle of the Method

Water samples received are analysed by operation of a Guildline Portasal Salinometer Model 8410A. The salinometer is designed to make precision conductivity comparisons between an unknown water sample and a reference sample. The results can be displayed as either Conductivity Ratio or Practical Salinity Units. Marine Scotland records PSU.

3. Reference Material

IAPSO Standard Seawater is the only internationally recognized calibration standard for the measurement of Practical Salinity as approved by all the major oceanographic bodies (ICES, IOC Unesco, SCOR,etc) and is endorsed by the International Association for Physical Sciences of the Ocean (IAPSO).

The current definition for Practical Salinity states: a seawater of Practical Salinity 35 has a conductivity ratio of unity at 15 degrees Centigrade (and 1 atmosphere pressure) with a potassium chloride (KCl) solution containing a mass of 32.4356 grams of KCl per kilogram of solution.

4. Reagents

Not applicable

5. Equipment

Guildline Portasal Model 8140A

6. Environmental Control

6.1 Salinometer Room
Before any samples are to be analysed or the machine set up, the air conditioner in the salinometer room/container should be set to maintain the room at the desired temperature – normally 19°C, this should be 2ºC below the bath temperature (usually set at 21ºC) of the salinometer.

6.2 Bottle cleaning See SOP 0220

After analysis of salinities, used salinity bottles complete with black outer cap are sent to room 506 for cleaning. Cap Inners. Cap inners are retained in the salinometer room. *See Insert Cleaning*

During the bottle washing process the black outer caps are removed, the bottles are put in the glass washer individually, upside down. The caps are washed separately. When the caps are put back on the bottles any bottles that are chipped or damaged are turned upside down, in the crate, for OS staff to check.

When the crates of clean bottles are returned:

- Clean off any previous cruise code or site name from the white identification plate using isopropanol cleaner.
- Check any upside down bottles to see if they can be safely used again; if not replace them and place the bottle in the waste glass box.
- Put a bag of 25 clean inserts, and 1 spare cap, into the crate
- Place in the designated position on shelf (or next to shelves)

Clean crates must be kept separate from the analysed crates ready for washing to avoid used (un-cleaned) bottles being sent out or put on the shelves.

6.2.1 Insert cleaning

Used or left over inserts are washed before re-use. Plastic bags that are returned may be discarded. The inserts and spare caps are first soaked and washed in tap water, preferably overnight, and then rinsed twice in distilled water. Drain off the water and spread the inserts over blue towelling. Then put a layer of blue towel over the inserts to stop dust settling on them.

Inserts are put in a new plastic bag, in a set of 25 for the crates or 12 for the LTM boxes, with a spare cap.

6.3 Crate maintenance

Crates returned from washing should be checked for damage. The lids should be hinged with three regular cable ties and kept shut with a single re-usable cable tie. On the lid, electrical tape with an arrow indicates the first bottle when the lid is open.
The identification plate on the front of the crate should be held on with four cable ties and display the bottle number range for the crate. Replacement plates can be made by the workshop and spares are kept in the wooden drawers opposite the shelving unit.

7. Interferences

Not Applicable

8. Sampling and Sample Preparation

8.1 Issue of Salinity Bottles

Salinity bottles within Marine Scotland Science are of the following design: 200ml bottles fabricated from Type II glass and utilizing two a two part cap. These maintain the integrity of the sample by protecting it from chemical and physical changes.

In general bottles are stored in wire crates of 24 bottles and are released as whole sets of 24. Bottles are numbered 1 - ~1000

Plastic bags with 25 inner and 1 spare outer black cap are prepared and kept in the Biscuit Box. These plastic bags are then placed in the full bottle crates when the bottles are clean and stored in their designated shelf position within the salinometer room.

For Long Term Monitoring sites at Scalloway (Shetland [ZE]), Scapa (Orkney [OR]) and Lochmaddy (Uist [LM]), as only twelve bottles are required per dispatch, these bottles are stored separately on the shelf above the salinometer, in the salinometer room (A11) and are numbered ZE / OR / LM 1-24.

Issue of all salinity bottles, whether general crates or LTM boxes, is entered in the appropriate Record Book - *person issuing, person issued to, project name/code, bottle number range and date of issue* - which is kept in the salinometer room. The cruise number or other recognisable designation, such as site name, is written on the white plate attached to the side of the crate.

8.2 Reception of Returned Salinity Bottles

(also nutrients & silicates from LTM)

Salinity bottles can come from a number of different sources, Scientific cruises, Fieldwork and Long Term Monitoring sites.

All bottles must be “Booked In” using the Record Book kept in the salinometer room, log the return date and, when completed, the date when of analysis. This is useful when tracing missing results or bottles.

Bottles must then be left in the salinometer room for at lease 24 hours in order to stabilize their temperature to that of the room before they may be analysed.

8.2.1 Nutrients and Silicates – (LTM)

Nutrient Bottles/Phials are stored in the freezer in 416

Silicate bottles/phials are stored in the fridge in 501B

Pam Walsham, Alison McIntosh and/or Alistair McIntosh are to be informed of their arrival.

9. Analytical Procedure

9.1 Standardization of the Salinometer Part I

If the salinometer is not powered up do the following:

Switch on the machine. Ensure there is no sample in the cell.

Select the bath temperature. This should be 2ºC above the room temperature.

To set the bath temperature

Press **T set** - Display reads **SET POINT TT**, Where TT in ºC will be flashing.

Press **SHIFT**, then, enter the bath temperature required.

Press **ENTER.** Press **ENTER**.

The bath temperature can take several hours to reach the set temperature. Therefore, it is advised to wait until the **NEXT DAY** before salinometer is ready for Standardization.

The set bath temperature is achieved when the heater lamps are flashing.

Salinity samples should be left in the salinometer room at least 24 hours to allow them to stabilize to the set room temperature.

NEXT DAY

Check the heater lamps. Are they flashing?

Yes – continue to ⇨**ANALYSING A WATER SAMPLE (For Standardization) 9.2.2**

Else – Set the bath temperature to 36ºC as above. If the bath water temperature is below this setting the heater lamps will light. If either of the heater lamps has failed the message, **HEATER 1 FAILURE** or **HEATER 2 FAILURE** will be displayed within 15 seconds.

Set the bath temperature to 15ºC as above. Observe that both heater lamps will remain off if the bath water temperature is above 15ºC.

Set the bath temperature to that set previously (usually 21ºC).
Wait for a minute.

Press **TEMP**

The display TEMP tt.tttºC must be within 0.02ºC of the set temperature.

Press ↑ **TH1** TEMP tt.tttºC will be displayed.

Press ↑ **TH2** TEMP tt.tttºC will be displayed and must be within 0.04ºC of **TH1** TEMP

Press ↓ twice to display TEMP in ºC.

If any of these checks fail see the Guildline Technical Manual for MODEL 8410A PORTASAL, Section 3.

9.2 STANDARDISATION OF THE SALINOMETER Part II

Ensure the cell is empty. The cell should be evacuated if it contains a sample. To do this press your finger on the **FLUSH** air vent. The **FLOW RATE** switch should be in the **OFF** position.

The Reference Calibration

Set **FUNCTION** switch to **STDBY**.

Press **REF** key

After approximately 8 seconds the display will read **-REFERENCE xxxxx** and update for 16 seconds. For example, xxxxx = 19892.
After a further 7 seconds the display will read **+REFERENCE xxxxx** and update

for 8 seconds. For example, xxxxx = 19892.
This cycle should be left to continue until you are satisfied that the **-REFERENCE** and **+REFERENCE** values are within ±2 counts and remain stable over 10 minutes.

Press **COND** key

The Zero Calibration

Set **FUNCTION** switch to **ZERO**

Press **COND** key

When the zero conductivity ratio measurement is stable, press **ZERO** key.

Display reads **ZERO x.xxxxx**, for example, x.xxxxx 0.00000, 0.00029, etc. This value should not exceed ±0.00075.

If satisfied that this number is not drifting press the **COND** key.

Display should then read **RATIO 0.00000**

If this reading is not stable either restart at **The Reference Calibration** or wait another half day as the bath temperature may not have stabilized.

Set **FUNCTION** switch to **STDBY**. Note the Ratio reading, example 1.32348.

9.2.2 ⇨ANALYSING A WATER SAMPLE (For Standardization)

Start from here if the salinometer has been left on and has gone through the above STANDARDIZATION procedure.

Using a bottle of IAPSO Standard Sea Water, fill the cell, flushing 3 times. If using the external sample pump, use setting 4.

Reduce the flow rate to a minimum.

Set **FUNCTION** switch to **READ**

Once the following steps commence DO NOT flush the cell and DO NOT move the **FUNCTION** switch from **READ**

Press **STD** key. Display reads **STD STANDARDIZE**. Press **ENTER** key.

Display prompts **COND NO x.xxxxxx**

Alter x.xxxxx value to that on the label of the IAPSO Standard Sea Water bottle. (to enter a decimal point, press **SHIFT 2** keys down together).

Press **ENTER** key

Display prompts **BATCH NO xxxx**

Alter xxxx to the batch number on the label of the IAPSO Standard Sea Water bottle. (e.g. P115, for a letter press **SHIFT** down with either ↑ or ↓).

Press **ENTER** key

Turn **FLOW RATE** to a suitable flow rate, ensure cell is full & sample flowing.

Display reads **ENTER WHEN READY**

Press **ENTER** key

When satisfied that the measured standardization is stable, for example **STANDARD x.xxxxx**, note the **STANDARD** value on the sheet and press **ENTER** key

The conductivity ratio will then be displayed and it should match that shown on the label of the IAPSO Standard Sea Water bottle.

Set **FUNCTION** switch to **STDBY**

Record the conductivity ratio, if this value changes significantly the instrument must be re-standardized.

After use, ensure that the IAPSO Standard Sea Water bottle insert is replaced.

The salinometer is now ready to analyse samples.

If the machine is to be left for a less than 12 hours leave the sample bottle in the sample bottle holder, set the **FUNCTION** switch to **STDBY** and turn off the **FLOW RATE**.

If the machine is to be left for over 12 hours, set the **FUNCTION** switch to **STDBY** remove the sample bottle and fill the cell with distilled water flushing at least 3 times, leave the cell full and turn off **FLOW RATE**.

9.3 Analysing samples

1. Remove the standardized seawater bottle or Distilled Water bottle from the tubing.
2. Wipe the tubing with Blue Towel
3. Before opening, rock the salinity bottle to eliminate gradients. Wipe the top of the next sample bottle, making sure not to let any salt crystals get into the sample bottle and attach to the tubing.
4. Flush the conductivity cell three times. Make sure there are no bubbles in the cell. If there are, flush again.
5. While flushing, gently shake the next sample bottle.
6. After the third flush, when the cell is filled, take a reading by turning the function switch to READ (the conductivity ratio will be displayed), press the SAL key. Record the result on the log sheet. Set **FUNCTION** switch to **STDBY** before emptying the cell.
7. Flush the cell again, and take another reading. Usually wait 5 seconds for the reading to stabilize before recording it. A large difference in the reading will indicate that one reading is incorrect, thus a third reading should be taken and an average of the two (close) readings recorded.
8. Remove the sample bottle, empty the bottle, replace its cap and return it to its crate.
9. Wipe the tubing down with blue towel.
10. Repeat steps 2-9 for each sample. Prior to taking a sample wipe the tube with a clean tissue.

Continue analysing samples by repeating the above instructions until all the salinity samples in the crate have been analysed.
Check for and discard any broken/chipped/cracked bottles/inserts/caps.
Note any cracked bottles or missing inserts on the Log Sheet.

At the end of each crate using water from the IAPSO Standard Sea Water bottle check that this waters conductivity ratio has not drifted. If there is any *significant* change in the conductivity ratio, open up another bottle of IAPSO Standard Sea Water and check again. If there is still signs of drifting the salinometer will need to be re-standardized. Go to **STANDARDIZATION OF THE SALINOMETER.**
If the problem persists there is likely to be a machine fault, cease analysis operations and seek further advice.

10. Calculation of Results

Not Applicable

11. Precision, Bias and Limit of Determination

The Guildline Portasal Model 8410A has a measurement range from 0.004 mS/cm to 76 mS/cm with a resolution of 0.0003 mS/cm. The measurement can be made at a bath temperature selectable from 15°C to 38°C in steps of 1°C the accuracy of the instrument is better than 0.003 equivalent Practical Salinity Units.

12. Reports

Processing, Communication and Diffusion of Results

12.1 **For LTM sites**, after analysis, results are entered into Microsoft Excel® and saved in the appropriate directory.

Salinity results from projects such as the Ecosystems sampling and WFD (Water Framework Directive) sampling are e-mailed to Susan Robinson (Loch Ewe and Stonehaven) and Alistair McIntosh, (Loch Torridon, Loch Ewe and Stonehaven).

The sheet is then filed in the green LTM 2.5 folder.

12.2 Salinity results from cruise CTD calibrations are entered onto the appropriate Environmental Station Record Sheet. The sheets are then sent, with a Data Preparation Request form to Diane Spalding in Room 213 and the data is typed-up and compiled into .pre and .sor files and saved in nts2:\hydro\Data_General\Typed Data\.

13. Safety

Refer to risk assessment RA OS 05

14. Literature References

Technical Manual TM 8410 A, Kept in blue box with salinometer spares.

UNESCO: The Practical Salinity Scale 1978 and the International Equation of State of Seawater
1980, UNESCO Technical Papers in Marine Science, 36, 25 pp., 1981

UNESCO: Algorithms for computation of fundamental properties of seawater, UNESCO Technical Papers in Marine Science, 44, 53 pp., 1983
www.teos-10.org Thermodynamic Equation of Seawater - 2010