Hg-free determination of heavy metals

Daily routine for best performance

Metrohm

Content

1 Introduction

The Hg-free VA daily routine is a document that outlines a specific series of actions for preparing an analytical system, conducting voltammetric determinations with Hg-free sensors as well as for storing the equipment.

The recommendations summarized in this document are applicable to all Hg-free methods and sensors, as well as to manual, semi-automated, and fully automated systems.

This document is divided into following parts:

1) Daily Routine – serves as a guide for the working day. This part collects and describes all steps that should be taken when starting up the system, running and shutting it down.
2) General Tips – in this chapter general information related to maintenance, storage and handling of dosing units, electrodes and software are summarized.
3) Troubleshooting Guide – this part lists typical problems, their potential sources, introduces possible solutions and answers FAQs.

The recommendations and descriptions in the following mainly refer to the analysis with a fully automated system. For the analysis with a manual system some recommendations may not apply.

The following documents also contain useful information and are referred to in this document, where applicable.

Manual – 884 Professional VA (8.884.8003EN)

Manual – 858 Professional Sample Processor (8.858.8002EN)

Manual – 843 Pump Station with membrane pump (8.843.8001EN)

Manual – 807 Dosing Unit (8.807.8002EN)

viva Help – Access in the **viva** software by pressing «F1» on the keyboard

Manual for **viva** 3.0 (8.0103.8001EN)

1.1 Checklist – Procedures at the beginning of the working day

The following steps should be carried out at the beginning of the working day before the first sample is determined.

	Procedures at the beginning of the working day
☐	Prepare new solutions according to instructions in specific application documentation (if needed).
☐	Plug in all system components (884 Professional VA instrument, sample processor and rinsing station) and adjust the contact pressure lever in the tubing cartridge
☐	Start **viva** software
☐	Fill rinsing canister with rinsing solution and all dosing units air-bubble-free with respective solutions
☐	Prepare the Hg-free sensor according to the instruction in specific application document
☐	Place the Hg-free sensor together with reference and auxiliary electrode in measuring head
☐	Activate and/or clean the sensors according to the instruction in specific application document
☐	Use a check standard solution to condition the sensor and to verify that the system is operating within an acceptable range.

1.2 Checklist – Procedures during the working day

The following steps should be carried out during the working day after finishing the test measurements in a check standard solution.

	Procedures during the working day
☐	Prepare samples and setup a sample table with all necessary information
☐	Analyze samples
☐	Monitor the analytical performance of the Hg-free sensor

1.3 Checklist – Procedures at the end of the working day

The following steps should be carried out at the end of the working day after the last sample was analyzed.

	Procedures at the end of the working day
☐	Rinse electrodes and measuring vessel
☐	Store the electrodes accordingly
☐	Fill all dosing units with ultrapure water
☐	Plug off all system components (884 Professional VA instrument, sample processor and rinsing station) and release pressure from the pump tubing of the sample changer

2 Startup – Procedures at the beginning of the working day

2.1 Prepare fresh solutions

Actions

- Rinse the glassware thoroughly multiple times (>3) with ultrapure water before and after preparation of solutions.
- Prepare all solutions (supporting electrolyte, check standard solution, standard solution and so on) required for the determination according to the specific application documentation.

Comments

- Use purest chemicals for the preparation of solutions required for the analysis if not otherwise stated in application documentation. It is recommended to use reagent grade «for trace analysis» or comparable.
- It is recommended to always use the same glassware for the same solution e. g. same bottle for the supporting electrolyte and so on.
- For storing, fill the glassware with storing solution (ultrapure water acidified with concentrated nitric acid (1 mL / 1 L)).

2.2 Plug in the 884 Professional VA instrument and prepare other system components

Actions

- Plug in the 884 Professional VA instrument and all peripheral devices (like autosampler or rinsing station if available).
- Fix the tubing cartridge so that the snap-action lever (1) snaps in. Adjust the contact pressure lever (2) that sample is transferred by the peristaltic pump.

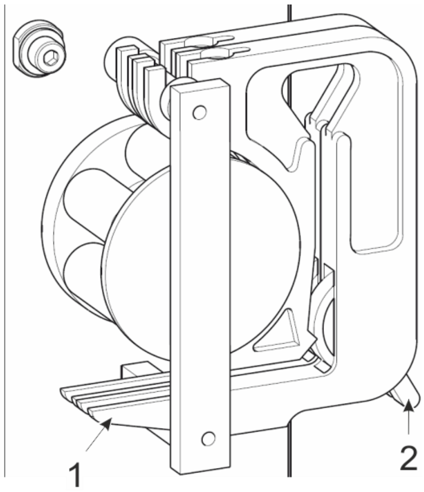


Figure 1 Tubing cartridge of the peristaltic pump of the 858 Professional Sample Processor

Comments

- Do not tighten the contact pressure lever (2) too much. This could block the pump and will reduce the lifetime of the pump tubing.

2.3 Start viva software

Actions

- Switch on the PC and start the **viva** software.

Comments

- It is recommended to use the newest version of **viva** software.

2.4 Fill rinsing canister with rinsing solution and all dosing units air-bubble-free with respective solutions

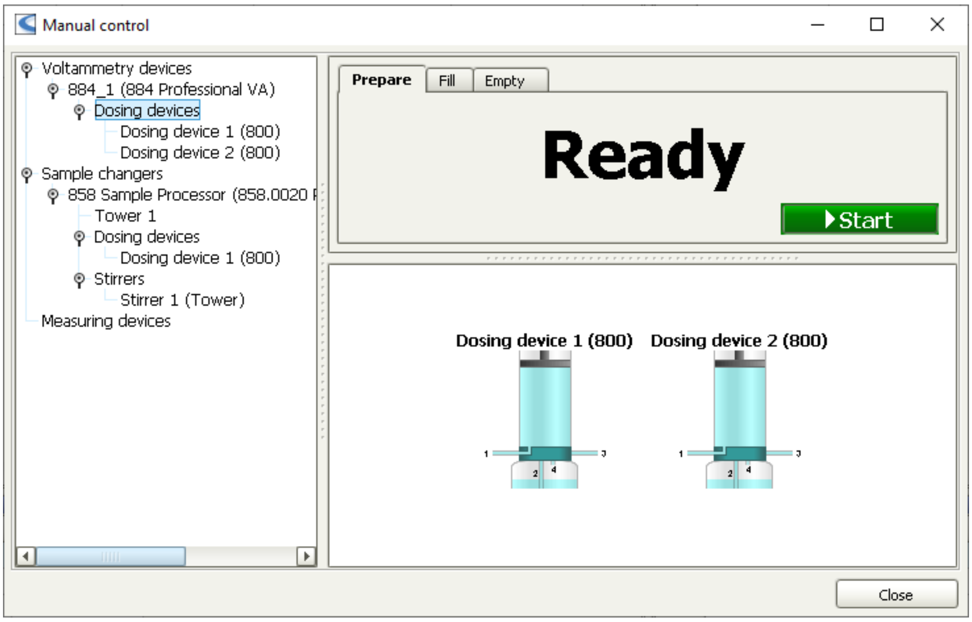
Figure 2: Container 10 L

Figure 3: 807 Dosing Units

Actions

- Use ultrapure water acidified with concentrated nitric acid (1 mL / 10 L) as a rinsing solution.
- Fill up the 10 L rinsing canister with the rinsing solution.
- Empty the waste canister if necessary.
- Check all tubings if they are tightly connected to the pump station and to the canisters.

- Position the newly prepared solution directly beneath the corresponding dosing unit and ensure that the aspiration tube is immersed in this solution.
- Prepare every dosing unit used during the determination with the respective solution at least 2 times using the «prepare» function in manual control in the **viva** software or alternatively using the automated method for system preparation.



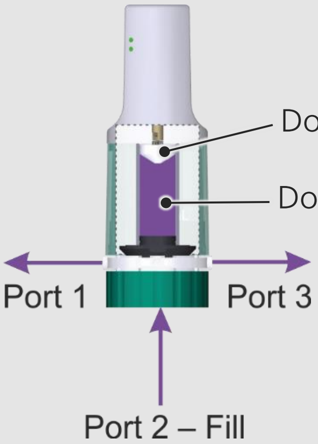
Manual control

- Voltammetry devices
  - 884_1 (884 Professional VA)
    - Dosing devices
      - Dosing device 1 (800)
      - Dosing device 2 (800)
- Sample changers
  - 858 Sample Processor (858.0020 P
    - Tower 1
    - Dosing devices
      - Dosing device 1 (800)
    - Stirrers
      - Stirrer 1 (Tower)
- Measuring devices

Figure 4: Manual control in the **viva** software

- Check if there are no air bubbles in the dosing cylinders.

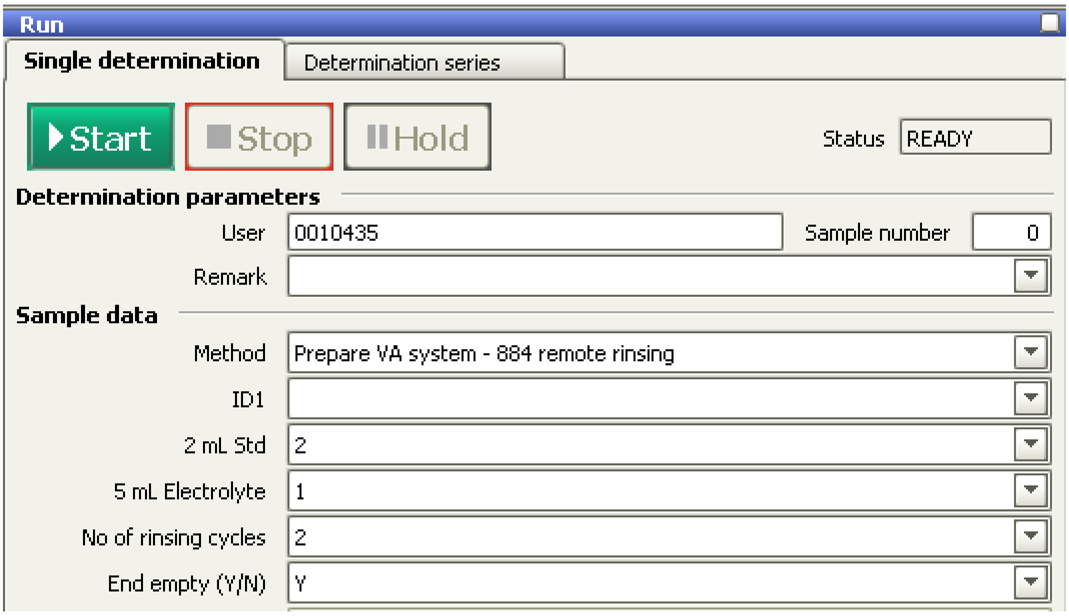
Info: *Why is the correct preparation of dosing unit(s) important?*



The dosing system consists of aspiration tube (port 2), dosing cylinder, dosing tube and capillaries (port 1 and 3). In order to correctly dose the volumes defined in the method all tubings and the dosing cylinder have to be completely filled air-bubble-free.

Comments

- All tubes and canisters should be clean.
- The preparation of the dosing units can be simplified if a method is used for the preparation, e. g. «Prepare VA system – 884 remote rinsing». In this method only the number of preparation cycles for each dosing unit (e.g. 2 mL Std, 5 mL Electrolyte) needs to be defined in the «Run» subwindow. If a dosing unit should not be prepared with solution the value «0» has to be entered. For more information about this method see Application Work AW VA CH-0627-022024.



Status READY

Determination parameters

User 0010435
Sample number 0
Remark

Sample data

Method Prepare VA system - 884 remote rinsing
ID1
2 mL Std 2
5 mL Electrolyte 1
No of rinsing cycles 2
End empty (Y/N) Y

Figure 5: Example for the «Run» subwindow with the method for the preparation of 807 Dosing Units

- In order to fill all tubings connected to the dosing unit automatically the length and the diameter of the tube have to be defined correctly in the configuration of the respective dosing unit in the **viva** software.
- A small air bubble can always remain at the piston, even after preparation of the dosing unit. This will not affect the accuracy of the dosing. Problems can only occur when the air bubble gets bigger and covers significant area (> 50%) of the dosing piston.

2.5 Prepare the Hg-free sensor according to the instruction in the specific application document

The term «Hg-free sensor» refers to a working electrode that does not utilize liquid mercury. For the analysis of heavy metals the glassy carbon and the Bi drop electrode require an external reference and auxiliary electrode. Platforms such as scTRACE Gold and 11L SPE integrate all three electrodes (WE, RE, and AE) on a ceramic support, offering a more convenient solution. A manual preparation (e. g.: polishing) is only required for the glassy carbon electrode. Hg-free sensors, reference electrodes, and auxiliary electrodes should not be cleaned using sonication with ultrasound or prepared with any other methods unless stated in the specific application documentation.

2.5.1 Glassy carbon

Actions

- Polish the glassy carbon electrode with the polishing powder provided by Metrohm according to the instructions in the electrode leaflet or in the application documentation.
- Clean the surface of the glassy carbon electrode with copious amount of ultrapure water after polishing.

Figure 6: Glassy carbon (GC) working electrode 6.1204.600

Comments

- After polishing, a mirror-like surface is expected.

2.6 Place electrodes in the measuring head

Actions

- Introduce required electrodes into the measuring head.
- Rinse all electrodes with ultrapure water.
- Prepare (activate, clean or modify with a metal film) the Hg-free sensor according to the specific application documentation.

Comments

- When positioning the electrodes in the measuring head, it is crucial to ensure that all electrodes are immersed in the solution, with particular emphasis on the complete immersion of the working electrode.
- No bubbles should be attached to the electrodes.

2.6.1 Activation/cleaning procedure (Bi drop, scTRACE Gold)

Actions

- Activate or clean the Hg-free sensor according to the specific application document.
- Rinse all electrodes and measuring vessel with ultrapure water.

Comments

- Do not clean or prepare the electrode with any other methods unless stated in the specific application documentation.
- Multiple activation of the scTRACE Gold decreases the lifetime of the sensor.
- The Bi drop electrode can be activated multiple times without effect on the lifetime.

Figure 7: Bi drop electrode (left), 11L SPE (middle) and scTRACE Gold (right)

2.6.2 Film deposition (Glassy carbon electrode, scTRACE Gold and 11L SPE)

Actions

- Carry out a film deposition procedure to the Hg-free sensor (Glassy carbon electrode, scTRACE Gold and 11L SPE).
- After finishing the film deposition procedure rinse thoroughly all electrodes and measuring vessel with ultrapure water.

Comments

- Do not clean or prepare the electrode with any other methods unless stated in the specific application documentation.
- In many cases, the plating solution can be reused several times. To facilitate this, collect the plating solution after completing the film deposition step. It is advisable to differentiate between new and used plating solutions by storing them separately.

2.7 Use a check standard solution to condition the sensor and to verify that the system is operating within an acceptable range

Actions

- Load the determination method and perform 3–5 determinations in a check standard solution.
- Rinse the electrodes and the measuring vessel with ultrapure water.
- Compare the obtained voltammograms with the voltammograms from the application documentation to identify significant differences.

Comments

- Determination in a check standard solution should be carried out immediately after the sensor preparation (activation, cleaning or plating of a metal film) step.
- For comparison absolute current values at specific potential, the shape of voltammograms, the slope of calibration curve (same deposition time important!) and the recovery rate of the spike can be employed.
- If nothing else is specified, the recovery rate of the test determination being in the range between 90% and 110% is acceptable.

3 Procedures during the working day

3.1 Prepare the samples and set up a sample table with all necessary sample information

Actions

- If required prepare the samples according to the specific application documentation (e. g. UV digestion, extraction).
- Dilute the samples if necessary to fit into the linear range of the method and to reduce the matrix effect.
- Accurately add the sample amount into a clean sample vessel on the sample changer or directly into the measuring vessel.
- Place the sample vessel on the rack of the sample changer if an autosampler is used.
- Load the determination method.

- Enter the sample ID and other necessary information in the sample table in the **viva** software.
- Enter the effectively added sample amount in the respective field in the sample table in the **viva** software.

Comments

- For sample preparation proceed according to the specific application documentation.
- Determinations are carried out in the order in which they are listed in the sample table. Individual samples can be excluded from the series, without deleting the line, using the function «Set lines inexecutable».
- By default, the text fields ID1 to ID3 are available to identify samples in the database and enter additional sample information. If necessary, additional fields can be activated in the method.
- Sample tables can be saved and loaded. For details see «**viva** Help – Support / How to proceed? / Sample tables».
- Reoccurring entries for sample data variables can be stored as text templates. This allows to simply select the entry for a certain ID instead of typing it in.

3.2 Analyze samples

Actions

- Start the determination method and proceed according to the specific application documentation or the instructions from the **viva** software.

Comments

- Sample analysis should be carried out directly after finishing measurements in the check standard solution.
- High reproducibility and repeatability can only be achieved when the sensor is continuously used.
- The interruption times between the sample determinations should be as short as possible.
- To ensure high reproducibility across all samples, it is essential that the sequence of events and the time intervals during and between subsequent determinations are identical. This can be achieved by using an automated system. However, when a manual system is employed, standard additions must be made at equal time intervals. Moreover, sensors should not be left unused for an undefined amount of time, and the same volume of rinsing solution should be used.

- During determination electrodes must not be removed from the measuring solution. The measuring head must not be lifted during a determination for this purpose. Additions have to be done via the pipetting opening.

3.3 Monitor the analytical performance of the Hg-free sensor

Actions

- To evaluate the matrix effect, compare the absolute current values at a specific potential, the shape of the voltammograms, and the slope of the calibration curve (noting that the same deposition time is crucial) obtained in the check standard solution with those in the sample.
- To evaluate the robustness of the method in specific sample perform 3 determinations in the same sample and monitor changes in the absolute current values at a specific potential, the shape of the voltammograms, and the slope of the calibration curve.

Comments

- If the sensitivity of the analytical method (slope of the calibration curve) drops below 50% compared to the sensitivity in a check standard solution the sensor should be cleaned according to the specific documentation and the sample should be diluted for future analysis (if possible).
- There are several tools that can be used to check correctness of the results, for instance: certified reference material (CRM) or spiking.
- CRMs are samples with known concentration of the analyte. They can be used to assess the accuracy and precision of the system. By measuring certified reference material at regular intervals throughout the day, any significant deviations or trends in the measurements can be detected, indicating a possible misbehavior in the system.
- Spiking the real sample with a known amount of analyte allows assessment of the accuracy of the obtained results in the sample matrix. Repeating this step at regular intervals can help detect changes in analytical performance.

4 Procedures at the end of the working day

4.1 Rinse electrodes and measuring vessel

Actions

- Rinse the inner of the measuring vessel and all electrodes thoroughly with ultrapure water.

- Place the cleaning solution in the measuring vessel and follow the cleaning procedure described in specific application documentation.
- After completing the cleaning method, rinse the measuring vessel and all electrodes with ultrapure water.

4.2 Store the electrodes accordingly

To ensure the longevity and reliability of your electrochemical sensors, proper maintenance and storage are crucial. Hg-free sensors portfolio includes various electrode types, each with specific handling requirements.

Actions

- Rinse the Bi drop electrode and reference electrode with ultrapure water and remove from measuring head after finishing the determination series.
- Dry the Bi drop electrode on air for ca. 30 min without touching the surface of the bismuth drop and cover it with the protective cover.
- Store the reference electrode in KCl solution (c(KCl) = 3 mol/L). Proper storage of reference electrode is essential to maintain its reference potential. To achieve this follow specific guidelines as described in chapter 5.2.4.
- Other electrodes can be stored in measuring head.
- Keep all electrodes away from environments that could lead to contamination or physical damage.

Comments

- Glassy carbon electrode is robust and can withstand prolonged exposure to the experimental or rinsing solution without significant degradation. It can remain in the measuring vessel overnight. For longer period of time (>2 days), it is recommended to remove the glassy carbon electrode out of the measuring head and store it elsewhere.
- The auxiliary electrode can be left in the measuring head after the experiments. Mechanical damage should be avoided by lowering the measuring arm.
- The scTRACE Gold and Screen-Printed Electrodes (SPE) that integrate all three electrode types (working, reference, and auxiliary) into a single unit should be handled with additional care.
- The scTRACE Gold sensor should be removed from the measuring/rinsing solution when not in use for more than 2h, rinsed with ultrapure water, dried and stored with protective cap. Storing it properly ensures the gold surface remains clean and active for accurate measurements.

- SPEs are designed for few measurements and have to be replaced after 5 determinations anyway. Determinations should be carried out directly after each other. Freshly prepared SPE-sensor must not dry out.
- If the SPE holder is used it is recommended to disassemble the holder after finishing the determination series and to dry the silicone sealing and the PTFE spacer with a tissue to prevent corrosion of electrical contacts inside the holder.
- For reference electrodes ensure the liquid level is properly maintained in the storing vessel, and the solution is not contaminated.
- If needed replace the inner and outer electrolyte of the reference electrode according to the table below.

Article no.	Reference electrolyte replacement intervals
6.0728.120	Once per month
6.1245.010	Once per week

- If the reference electrode (6.0728.120) should be refilled with 3 mol/L KCl electrolyte make sure that at least 4 hours are needed for the reference potential to equilibrate. Refilling the reference electrode at the end of the working day gives the electrode sufficient time for equilibration over night.
- If the maintenance-free reference electrode (6.0730.100) is used, no actions are necessary.

4.3 Fill all dosing units with water

Actions

- Prepare and fill all used dosing units with ultrapure water at least 2 times by using the «Prepare» function in **viva** «Manual control » or by using the dedicated method for preparation of dosing units mentioned in chapter 2.4.

Comments

- Storing the dosing unit with water prevents blockage of the valve and the formation of crystals inside the dosing cylinder.
- Ideally, the preparation process should not involve placing the measuring vessel under the measuring head. A waste beaker can be positioned below the measuring head to prevent carry over effect.

- When the dosing unit is only emptied, a small amount of solution will remain in the dosing cylinder and in the tubings. In this way the solvents can evaporate more easily, thus facilitating the formation of crystals.

4.4 Release pressure from the pump tubing of the sample changer

Actions

- When the peristaltic pump for sample transfer is not used for a longer time, e. g., over the weekend, it is recommended to release the pressure from the pump tubing by unlocking the snap-action lever (1).

Comments

- It is recommended to leave the contact pressure lever (2), which is used for the adjustment of the flow rate in position. In this way the peristaltic pump can easily be set back to work by fastening the snap-action lever.

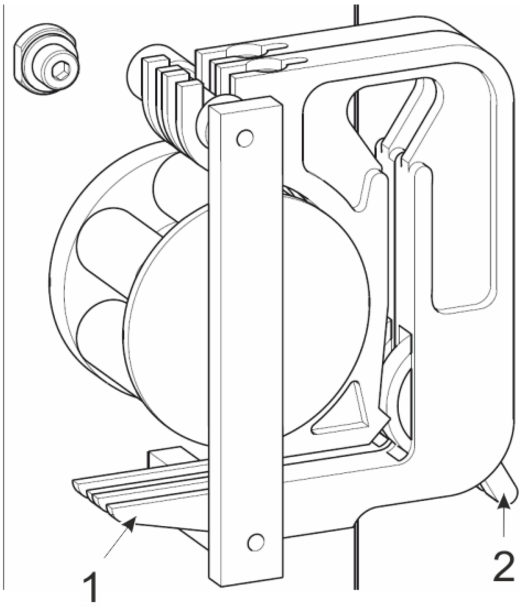


Figure 8: Tubing cartridge of the peristaltic pump of 858 Professional Sample Processor

Info: *Why should the pressure be released from the pump tubing?*

By releasing the pressure when the pump tubing is not in use the lifetime of the tubing can be increased. When the pump tubing stays clamped while it is not in use it can deform. This does not only affect the efficiency of the pump but can also fatigue the material making it more prone for fracturing.

5 General tips and information

5.1 807 Dosing Units

5.1.1 807 Dosing Unit configuration

For 807 Dosing Units it is recommended to use the following setup, where port 3 is used for the preparation.

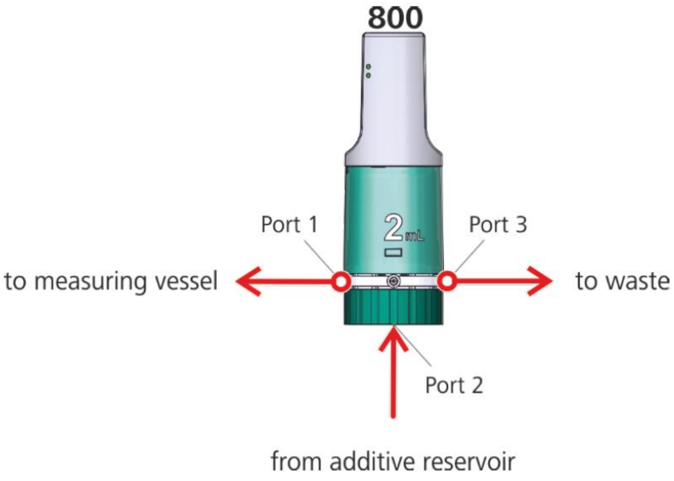
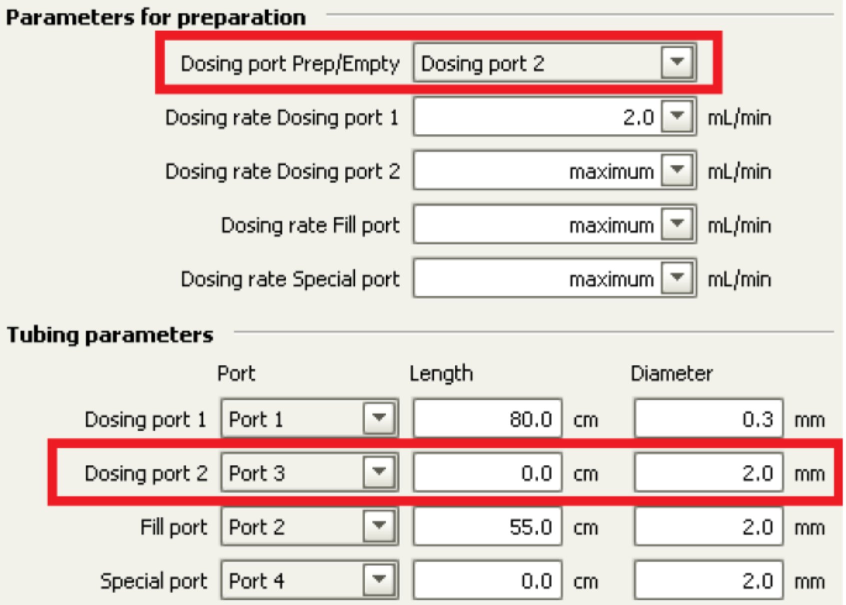


Figure 9: 807 Dosing Units tubing configuration for preparation via port 3

Info: *What is the benefit of preparing the 807 Dosing Units via port 3?*

- Faster: Since an FEP tubing of 2 mm inner diameter can be used on port 3 the dispensing of solution to the waste is much faster than into the measuring vessel via e. g. a capillary of 0.3 mm inner diameter as used in the four-way micro dosing tip (6.1824.000). Smaller diameters limit the dosing speed.
- Less contamination: If the preparation is done via port 1 a very high volume of the respective solution goes into the measuring vessel. This can result in memory effects if rinsing is done insufficiently.

The use of port 3 for preparation needs to be defined in the software. In the **viva** software this setting is found under the properties of the respective 807 Dosing Units. Note that the length of the waste tubing should be specified as 0 cm to avoid that this tubing will also be completely filled during preparation.



Parameters for preparation

Dosing port Prep/Empty	Dosing port 2	
Dosing rate Dosing port 1	2.0	mL/min
Dosing rate Dosing port 2	maximum	mL/min
Dosing rate Fill port	maximum	mL/min
Dosing rate Special port	maximum	mL/min

Tubing parameters

	Port	Length	Diameter
Dosing port 1	Port 1	80.0 cm	0.3 mm
Dosing port 2	Port 3	0.0 cm	2.0 mm
Fill port	Port 2	55.0 cm	2.0 mm
Special port	Port 4	0.0 cm	2.0 mm

Figure 10: Settings for preparation via port 3 in the **viva** software

5.1.2 Maintenance of 807 Dosing Units

Comments

- Apart from rinsing with water, which is always recommended at the end of the working day (see chapter 4.3), the 807 Dosing Units should be checked and maintained in regular intervals.
- For further information on handling and maintenance of the 807 Dosing Units also check the Manual 807 Dosing Unit

5.2 Electrodes

5.2.1 Short time storage

- If the electrodes are not used for less than half an hour, they can remain immersed in the solution.
- The only exception are the SPEs. When SPE is used determinations should be carried out directly after each other without any interruptions to prevent the increase of the background current.

5.2.2 Long time storage

- If the electrodes are not used for more than half an hour, they must be removed from the solution and stored according to the procedure described in chapter 4.2.

5.2.3 Cleaning of electrodes

Mechanical cleaning of electrodes

- Do not treat any electrode (WE, RE or AE) or a combined sensor in an ultrasonic bath.
- Mechanical cleaning can only be applied to the glassy carbon electrodes (working and auxiliary electrode).
- The glassy carbon electrode can be cleaned mechanically by wiping it with a soft, lint-free tissue (e. g. Kimtech® Science precision wipes).
- If problems arise with mercury film deposition on the surface of the glass carbon electrode, follow these steps for cleaning:
    - Moisten a tissue with a more convenient solvent, such as isopropanol or ethanol.
    - Thoroughly clean the glassy carbon electrode with ultrapure water.
    - Optionally, polish the electrode.
- The glassy carbon electrode can be mechanically cleaned by polishing with aluminum oxide powder provided by Metrohm. After polishing the electrode has to be thoroughly rinsed, first with ultrapure water, then with ethanol or isopropanol and at the end once again with ultrapure water.
- Afterwards the electrode should be electrochemically conditioned/prepared according to the method described in specific application document.
- Finally, it is recommended to check the electrode performance as described in chapter 2.7.
- Additional information on the polishing of the glassy carbon electrode can be found on the Metrohm website www.metrohm.com in the section Support and Service > Product Help-Center > Voltammetry > Maintenance – Rotating Disk Electrode > Glassy Carbon RDE > 884 Professional VA > SEQ 2: How can I polish my glassy carbon electrode?
- The Bi drop electrode, the scTRACE Gold, the 11L SPE and reference electrode must not be cleaned mechanically.

Electrochemical cleaning of the working electrode

- The electrochemical cleaning should be carried out always: i) after the activation step, ii) when the sensitivity drops below 50%, iii) at the end of a working day / determination series and iv) whenever it is necessary.
- The electrochemical cleaning should be carried out according to the specific application documentation.

Criteria for the efficiency assessment of the electrochemical cleaning

- The simplest way to evaluate the efficiency of the cleaning step is to compare the sensitivity of a determination in a check standard solution before and after the cleaning step.
- Another option is to compare the recorded voltammogram with the one in the specific application documentation. For comparison typical parameters like sensitivity, absolute current value at specific potential or the shape of the background current can be employed.

5.2.4 Reference electrode

Figure 15: Reference electrode 6.0728.120

The KCl electrolyte ((c(KCl) = 3 mol/L)) in the reference electrode (6.0728.120) should be replaced once in a month.

After replacing the electrolyte allow at least 4 hours for the reference potential to equilibrate. Refilling the reference electrode at the end of the working day gives the electrode potential sufficient time for equilibration.

Care should be taken that no air bubbles are present at the diaphragm or in or around the AgCl packing to avoid contact problems.

More information on how to handle the reference electrodes in voltammetry can be found on metrohm.com. If the maintenance-free reference electrode (6.0730.100) is used, no actions are necessary.

5.3 Curve assessment

- The assessment of voltammetric curves typically involves comparing the shapes and characteristic parameters (e.g.: absolute current value at specific potential) of the voltammograms obtained in a check standard solution with those of the sample solution being analyzed. This comparison helps to identify any differences in the voltammetric response. Those differences can be used to estimate the degree of contamination or matrix effect within the sample. In addition, it can help to monitor changes of the system's performance over time.

- In this context, one of the most important parameters is the sensitivity, which is defined as the slope of the calibration curve.
- In some case the sensitivity may remain unchanged, but the recovery rate decreases due to changes of the background current which are affecting the sample peak intensity. Therefore, a monitoring of absolute current values for every peak is important part of the curve assessment procedure.

Actions

- Prepare the Hg-free sensor according to specific application documentation. Run 3 – 5 determinations in a check standard solution. Note the key parameters (shape of the voltammogram, peaks height, current value at specific potential and sensitivity) and monitor these values while conducting the sample series.
- If there are significant changes observable apply cleaning procedure or refer to specific application documentation to restore previous performance. In most of the cases, sample dilution can reduce the detrimental matrix effect.

6 Troubleshooting

6.1 Checklist

Please check the following points when problems occur (unusual voltammograms, no results, results outside the expected range, poor repeatability, etc.):

	Check	Action
☐	Have all electrode cables been connected correctly?	Connect all electrode cables correctly: Cable WE – Working electrode Cable RE – Reference electrode Cable AE – Auxiliary electrode
☐	Is the RE filled with the correct electrolyte and air-bubble-free?	Use the KCl electrolyte (c(KCl) = 3 mol/L) to fill the RE and make sure that there are no air bubbles inside the RE after refilling.
☐	Have all tubings been connected and are all tubing connections tight?	Check all tubings and connections. If needed, use the wrench 6.2739.000 to tighten the tubing connections.
☐	Have all 807 Dosing Units been filled air-bubble-free?	Repeat the preparation of the 807 Dosing Unit. If preparation of the 807 Dosing Unit does not remove the air bubble, check all tubing connections for tightness and repeat the preparation. If all connections are tight, and the air bubble remains, do a maintenance on the 807 Dosing Unit. If all measures do not solve the problem, replace the 807 Dosing Unit.
☐	Have all 807 Dosing Units been filled with the correct solution?	Double-check the used solutions. In case of doubts prepare fresh solutions and re-fill the 807 Dosing Units.
☐	Has the correct supporting electrolyte been used for the application?	Check in the method that the correct supporting electrolyte is used in the dosing command. Check in the configuration that the solution to be dosed is linked to the correct 807 Dosing Unit.

☐	Is the contact pressure for the peristaltic pump correctly adjusted?	Change the contact pressure and check whether that solves the problem.
☐	Are the pump tubings in proper condition?	Pump tubings are consumables. When alterations can be observed, e. g., fracturing in the sidewalls or a hardening of the material, the pump tubing should be replaced.
☐	Is the analyte peak correctly evaluated?	Check the voltammograms whether the correct peak is evaluated, and whether the baseline is drawn correctly. If needed, adjust evaluation parameters, such as the characteristic potential of the substance or the smoothing of the curve in the **viva** software.
☐	Is the specified sample amount correct?	Make sure that the sample amount specified in the determination corresponds to the sample size added into the measuring vessel
☐	Is the recovery rate in a check standard solution in the range of 100% ± 10 %?	Check the used method, check the standard solution.

6.2 FAQs

a) The preparation of the 807 Dosing Units is so slow. How can I speed it up?

The specified dosing rate has to be checked in the configuration. With an FEP tubing of 2 mm inner diameter the maximum dosing rate is the cylinder volume (in mL) multiplied by 3, which gives the dosing rate in mL/min. E. g. with a 2 mL dosing unit the maximum dosing rate is 6 mL/min. If a dosing capillary with an inner diameter of 0.3 mm is used the maximum permissible dosing rate is 4 mL/min. Our recommendation is to use 2 mL/min. Preparation can be done faster by using port 3, as described in chapter 2.4.

b) After a very short time there is a big gas bubble around the piston in the 807 Dosing Units. Is that normal? What can I do?

A small gas bubble at the piston is not unusual, even directly after the preparation of the 807 Dosing Units. A small gas bubble does not affect the correct dosing. When solutions with high vapor pressure, such as organic solvents used in the application, are dosed it is also not unusual that the gas bubble increases over time. It becomes problematic when a gas bubble gradually increases in size, and if the size of the gas bubble exceeds half of the piston area. To prevent the formation of a gas bubble with a critical size, it is recommended to always prepare the 807 Dosing Units at the beginning of the working day.

If the gas bubble exceeds half of the piston area within a few determinations, it is an indication that either the 807 Dosing Units leaks or one of the connections is not tight. In this case first check all the tubing connections. You may use the wrench 6.2739.000 to fasten all tubing connectors.

If that does not help, do maintenance on the 807 Dosing Units as described in chapter 5.1.2.

If also maintenance does not improve the situation, the 807 Dosing Unit is worn out. In this case the dosing cylinder or the complete 807 Dosing Unit has to be replaced.

c) Is it possible to use different acid than nitric acid for acidification of rinsing solution?

In general, it is not recommended to use other acids despite nitric acid for acidification due to possible interferences. However, in some specific applications (where the mentioned acid is used as an electrolyte) it can be possible to use HCl or H_2SO_4 for acidification.

d) How stable are the solutions used for my determination?

A detailed information regarding the stability of the solutions used for a determination can be found in specific application documentation. In general, the supporting electrolyte solution and standard addition solutions can be stored for a few days (if not otherwise stated) and the solution containing complexing agent or oxidation reagent for a day.

e) How often can I use the Hg-film plating solution?

The Hg-film plating solution can be used for ca. 10–15 plating events.

f) How many determinations can I do with my sensor?

The exact number of determinations depends on the application, sample matrix and the used sensor. With the scTRACE Gold a few dozens of measurements in a check standard solution are usually possible for most of the applications. For the SPE between 4–10 measurements are possible. A rough estimate of the number of possible determinations can be found in the specific application documentation.

g) Do you provide «ready-to-use» reagent kits?

Metrohm does not provide «ready-to-use» reagent kits. In order to keep the cost of determination as low as possible, we share our knowledge regarding the required chemicals and composition of solutions with our customers. In this way, the solutions can be prepared by our customers at significantly lower prices.

h) How often should I activate and clean my sensors?

Only the Bi drop electrode and the scTRACE Gold have to be activated before first use. Activation of scTRACE Gold is carried out only **once**, before the electrode is used for the first time. Activation of Bi drop is repeated multiple times in the lifetime of the electrode. Cleaning has to be carried out more frequently than activation. The frequency of cleaning depends on the application, number of analyzed samples, and the matrix.

i) Is it possible to accelerate the Hg-film plating step by reducing either the number of deposition cycles or deposition time?

No, any changes applied to the method for Hg-film plating will affect the properties of the mercury film. There is no guarantee that after modification of the plating procedure the determination will work as with original settings.

j) Is it possible to speed up the determination by reducing the number of additions and replication?

The provided methods are optimized for accuracy. This means sample and standard additions are measured in duplicate (2 replications) and 2 standard additions are carried out. If time is an issue, the number of replications and additions can be reduced to 1 each, reducing the analysis time to approx. 3–5 min. In this way the analysis will be faster but also less accurate.

k) Is it necessary to apply 300 s for purging or can it be shortened?

300 s are set as default values. If there is no dedicated instruction in application documentation to change this value, use the default value. Please refer to specific application documentation for more information.

l) How can I increase/decrease the sensitivity of the method.

This can be achieved by increasing/decreasing the deposition time.

m) How can I reduce the effect of interferences?

Depending on analyte concentration this can be achieved by diluting the sample with ultrapure water or by digesting the sample before analysis. Note that dilution may have an effect on accuracy.

6.3 Troubleshooting guide

Problem	Possible cause	Possible solution
Bad reproducibility of voltammograms	**Voltammetric parameters:**	
	Wrong voltammetric parameters	Use correct voltammetric parameters of application documentation
	Chemicals/cleanness of the system:	
	Contaminated solutions	Prepare new solutions Use purest chemicals for preparation of solutions Clean the glassware used for preparation and storage of all solutions
	No supporting electrolyte	Add all solutions specified in the application documentation into measuring vessel before starting measurement
	Matrix effect	Dilute the sample or use the 909 UV Digester
	Hardware:	
	Sensor not well conditioned	Repeat cleaning and 3–5 measurements in a check standard solution
	Electrodes wrongly connected	Connect all electrodes with the correct cables
	Air bubbles on the Sensor	Lift the electrodes out of the measuring solution after measurements is finished, rinse electrodes and repeat measurement
	Wrong RE filling	Rinse the RE with the appropriate solution three times, using approximately 5 mL each time, and then allow for equilibration.
	Air bubbles in the RE	Remove the air bubble from the RE
	Solution not degassed	Degas the measuring solution
	Stirrer vibrating	Replace the flexible drive shaft (6.1911.000) for stirrer. Make sure that it is correctly attached/inserted.

	Abrasion at the driving axle	Replace the driving axle
	Broken sliding contact	Replace the driving axle
	Lifetime of the sensor expired	Replace the sensor
	Bad condition of the sensor (scratch, damaged)	Replace the sensor
	Bad condition of electrodes	Replace electrodes
	Cables defective	Replace the defective cable
Noisy voltammograms	**Voltammetric parameters:**	
	Wrong voltammetric parameters	Use correct voltammetric parameters from application documentation
	Chemicals/cleanness of the system:	
	No supporting electrolyte	Add all solutions specified in the application documentation into measuring vessel before starting measurement
	Hardware:	
	Sensor not well conditioned	Repeat cleaning and 3–5 measurements in a check standard solution
	Electrodes wrongly connected	Connect all electrodes with the correct cables
	Air bubbles on the Sensor	Lift the electrodes out of the measuring solution after measurements is finished
	Wrong RE filling	Rinse the RE with the appropriate solution three times, using approximately 5 mL each time, and then allow for equilibration.
	Air bubbles in the RE	Remove the air bubble from the RE
	807 Dosing Units not grounded	Ground all 807 Dosing Units used in the analytical system
	Solution not degassed	Degas the measuring solution
	Stirrer vibrating	Replace the flexible drive shaft (6.1911.000) for stirrer. Make sure that it is correctly attached.

	Abrasion at the driving axle	Replace the driving axle
	Broken sliding contact	Replace the driving axle
	Lifetime of the sensor expired	Replace the sensor
	Bad condition of the sensor (scratch, damaged)	Replace the sensor
	Bad condition of electrodes	Replace electrodes
	Cables defective	Replace the defective cable
	Wet electrical contacts	Make sure that the contacts are clean and dry
Deviating shape of voltammogram	**Voltammetric parameters:**	
	Wrong voltammetric parameters	Use correct voltammetric parameters from application documentation
	Chemicals/cleanness of the system:	
	No supporting electrolyte	Add all solutions specified in the application documentation into measuring vessel before starting measurement
	Different composition or concentration of supporting electrolyte	Prepare all solutions accordingly to the application documentation
	Old solution	Prepare new solution
	Hardware:	
	Sensor not well conditioned	Repeat cleaning and 3–5 measurements in a check standard solution
	Air bubbles on the Sensor	Lift the electrodes out of the measuring solution after measurements is finished
	Wrong RE filling	Rinse the RE with the appropriate solution three times, using approximately 5 mL each time, and then allow for equilibration.

	Air bubbles in the RE	Remove the air bubble from the RE
	Solution not degassed	Degas the measuring solution
	Lifetime of the sensor expired	Replace the sensor
	Bad condition of the sensor (scratch, damaged)	Replace the sensor
	Bad condition of electrodes	Replace electrodes
	Wet electrical contacts	Make sure that the contacts are clean and dry
Unstable background current	**Voltammetric parameters:**	
	Wrong voltammetric parameters	Use correct voltammetric parameters from application documentation
	Chemicals/cleanness of the system:	
	No supporting electrolyte	Add all solutions specified in the application documentation into measuring vessel before starting measurement
	Different composition or concentration of supporting electrolyte	Prepare all solutions accordingly to the application documentation
	Hardware:	
	Sensor not well conditioned	Repeat cleaning and 3–5 measurements in a check standard solution
	Air bubbles on the Sensor	Lift the electrodes out of the measuring solution after measurements is finished
	Wrong RE filling	Rinse the RE with the appropriate solution three times, using approximately 5 mL each time, and then allow for equilibration.
	Air bubbles in the RE	Remove the air bubble from the RE
	807 Dosing Units not grounded	Ground all 807 Dosing Units used in the analytical system

	Solution not degassed	Degas the measuring solution
	Stirrer vibrating	Replace the flexible drive shaft (6.1911.000) for stirrer. Make sure that it is correctly attached.
	Lifetime of the sensor expired	Replace the sensor
	Bad condition of the sensor (scratch, damaged)	Replace the sensor
	Bad condition of electrodes	Replace electrodes
	Wet electrical contacts	Make sure that the contacts are clean and dry
Extremely high current values	**Voltammetric parameters:**	
	Wrong voltammetric parameters	Use correct voltammetric parameters from application documentation
	Chemicals/cleanness of the system:	
	Wrong supporting electrolyte / pH	Add all solutions specified in the application documentation into measuring vessel before starting measurement
	Hardware:	
	Electrodes wrongly connected	Connect all electrodes with the correct cables
	Air bubbles on the Sensor	Lift the electrodes out of the measuring solution after measurements is finished
	Wrong RE filling	Rinse the RE with the appropriate solution three times, using approximately 5 mL each time, and then allow for equilibration.
	Air bubbles in the RE	Remove the air bubble from the RE

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