Application Bulletin 800105021EN

Installation instruction: Professional CVS systems semiautomated

Semiautomated Professional CVS systems are used for the determination of additives in plating baths. These systems can automatically dose auxiliary solutions and standards.

Important features:

- Automatic addition of solutions like VMS, suppressor, brightener, leveler, or a plating bath sample.
- System is customizable with a flexible number of 807
 Dosing Units depending on how many solutions should be added automatically.
- Applications:
 - Determination of the suppressor concentration in plating baths with CVS or CPVS.
 - Determination of the brightener concentration in plating baths with CVS or CPVS.
 - Determination of the leveler concentration in plating baths with CVS or CPVS.

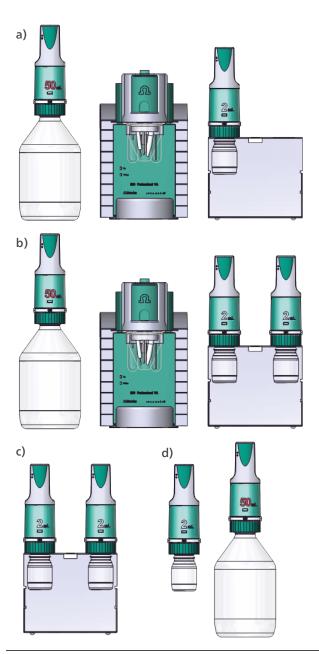


Figure 1. Semiautomated Professional CVS systems a) with two Dosinos (2.884.1210, 2.894.1210), b) with three Dosinos (2.884.1310, 2.894.1310) and optional equipment with 807 Dosing Units, c) 2 mL + 2 mL (6.5339.500), and d) 2 mL + 50 mL (6.5339.520)

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1 Instruments and accessories

Semiautomated systems are available for CVS applications with either the 884 Professional VA or the 894 Professional CVS with two or three 800 Dosinos. Also included are either two 807 Dosing Units with 50 mL + 2 mL (Figure 1a) or three 807 Dosing Units with 50 mL + 2 \times 2 mL (Figure 1b).

More solutions can be handled when the semiautomated systems are combined with 807 Dosing Unit equipment. These are available with 2 mL + 2 mL (**Figure 1c**) and 50 mL + 2 mL (**Figure 1d**) dosing units.

As an example, the setup with an 894 Professional CVS and four 807 Dosing Units (1 \times 50 mL and 3 \times 2 mL) is described in this document.

Quantity	Article no.	Designation
1	2.894.1210*	894 Professional CVS semi- automated 2 Dosinos
2	2.800.0020	800 Dosino
1	6.5339.500	Equipment with two 807 Dosing Units (2 mL + 2 mL) for VA/CVS
1	6.5339.xx0	CVS electrode equipment
1	6.6065.30x	viva 3.0

^{*} or 2.884.1210 (option with the 884 Professional VA)

Modifications and required accessories for the use of more than four 800 Dosinos are described in chapter 6.

2 System setup

2.1 Electrical connections

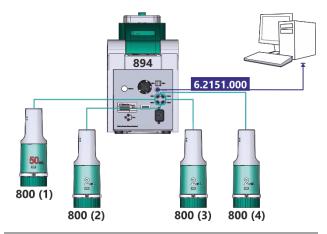


Figure 2. Electrical connections Professional CVS semiautomated.

2.2 Tubing connections

2.2.1 Overview

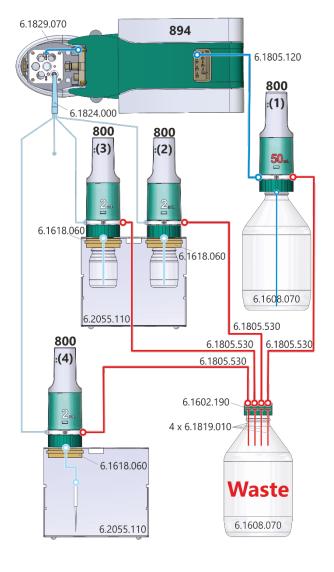


Figure 3. Example of tubing connections with four 807 Dosing Units $(1 \times 50 \text{ mL})$ and $3 \times 2 \text{ mL}$).

2.2.2 807 Dosing Units 2 mL

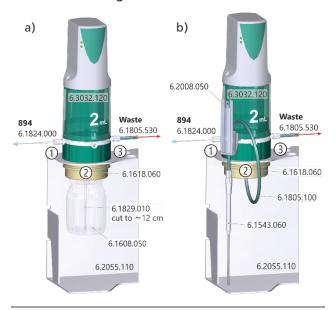


Figure 4. Options for the setup of tubing connections of the 2 mL dosing units used for addition of e.g., suppressor or brightener concentrate, a) with glass bottle if solutions do not need to be exchanged too often, and b) with tubing for easier exchange of solutions.

2.2.3 807 Dosing Unit 50 mL



Figure 5. Setup of tubing connections of the 50 mL dosing unit used for the addition of e.g., VMS.

⚠ Metrohm

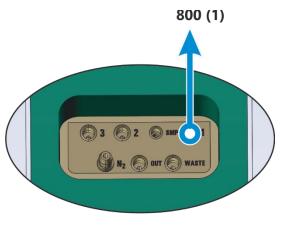
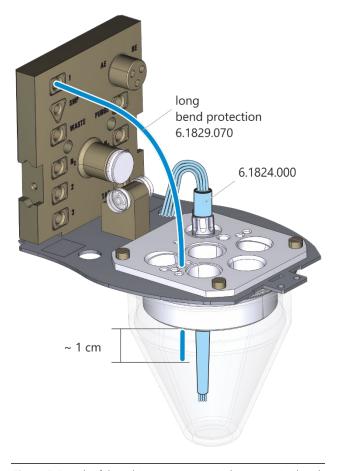


Figure 6. Details of the tubing connector located on the back of the measuring head arm.



 $\textbf{Figure 7.} \ \ \textbf{Details of the tubing connections in the measuring head}.$

3 viva «Configuration»



In addition to this Application Bulletin, it is recommended to have the following document available:

8.103.8010xx	viva Tutorial CVS
	In the following chapters, this document will be referred to as <i>Tutorial</i> .

3.1 Devices

The 894 Professional CVS is automatically recognized by the **viva** software. When an instrument is connected for the first time, it needs to be entered in the **viva** «Configuration». The corresponding dialog will pop up automatically.

Device name*	894_1
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*As used in the methods provided with this Application Bulletin.

For a step-by-step description, please read the *Tutorial* chapter «4.1.1 Configuring the instrument».

3.2 Sensors/Electrodes

A set of electrodes is already preinstalled in the **viva** software in the «Configuration». The following three electrodes must be present if the measuring command is used as specified in the **viva** example methods provided with this Application Bulletin.

Sensor name	Sensor type
Auxiliary electrode	Auxiliary electrode
RDE	RDE
Reference electrode	Reference electrode

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3.3 807 Dosing Units

An 807 Dosing Unit attached to an 800 Dosino which is connected to the 884 Professional VA is automatically recognized by the **viva** software. When the 807 Dosing Unit is connected for the first time, it must be entered in the **viva** «Configuration». The corresponding dialog will pop up automatically. For a step-by-step description, please see the *Tutorial* chapter «5.1.3 Configuring dosing units».

The following 807 Dosing Unit names correspond to the names used in the **viva** example methods provided with this document. However, 807 Dosing Unit names can be freely chosen, independent of instrument and MSB port the Dosino is physically connected to.

3.3.1 807 Dosing Unit 50 mL

Hardware

Name (1) 50 mL VMS

Parameters for preparation

Dosing port Prep/Empty	Dosing port 2	
Dosing rate Dosing port 1	maximum	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	138 cm	2 mm
Dosing port 2	Port 3	0 cm	2 mm
Fill port	Port 2	30 cm	2 mm
Special port	Port 4	0 cm	2 mm

Valve disk

Rotation direction	automatic
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3.3.2 807 Dosing Unit 2 mL

Hardware

Name (2)	2 mL Brightener
Name (3)	2 mL Suppressor

Parameters for preparation

Dosing port Prep/Empty	Dosing port 2	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 cm	0.3 mm
Dosing port 2	Port 3	0 cm	2 mm
Fill port	Port 2	12/55 cm*	2 mm
Special port	Port 4	0 cm	2 mm

Valve disk

Rotation direction	automatic
*Donands on the setup of the	207 Dosing Unit 2 ml (chan

^{*}Depends on the setup of the 807 Dosing Unit 2 mL (chapter 2.2.2): 12 cm for option a), or 55 cm for option b).

3.4 Solutions

Solutions that should be added automatically have to be defined in the **viva** «Configuration» and must be assigned to the 807 Dosing Unit which is used for the addition. For a step-by-step description, please see the *Tutorial* chapter «5.1.4 Defining solutions».

Table 1 shows the solution names and corresponding 807 Dosing Units as used in the **viva** example methods.

Table 1. 807 Dosing Units and solutions.

Solution name	Type*	807 Dosing Unit
VMS	А	50 mL VMS
Suppressor concentrate	А	2 mL Suppressor
Brightener concentrate	S	2 mL Brightener
Standard or sample	S	2 mL Standard or sample

^{*}Solution type: A = auxiliary solution, S = standard solution





This Application Bulletin comes with six example methods using the described hardware setup:

- 800105021EN Conditioning VMS semiautomated
- 800105021EN Brightener (CVS, MLAT) semiautomated
- 800105021EN Suppressor (CVS, DT) semiautomated
- 800105021EN Leveler determination (CVS, RC) semiautomated
 - With dilution
 - No dilution
- 800105021EN Preparation of dosing units

The methods already establish the basic sequences for the different calibration techniques, the use of 800 Dosinos for automatic addition, as well as the calculation of the result. However, certain commands and settings have to be adapted to the used hardware and the requirements of the specific application.

4.1 Method run

4.1.1 Measuring commands



- If the name of the 894 Professional CVS differs from the specifications in 3.1, then assign the correct instrument name.
- Adapt the measuring parameters on the tabs «Pretreatment», «Sweep», «Post-treatment», and «Potentiostat» according to the requirements of the application. These parameters can be found in separate application documentation.

4.1.2 Dosing commands

ADD STD	ADD STD
ADD STD	ADD STANDARD DT

- Select the solution and volume which should be added when this command is due.
- If the name of the solution differs from the specifications in 3.4, then select the correct solution name under Standard Solution in the properties of the command.

Please Note!

The name of solutions added with the 807 Dosing Unit «2 mL Standard or sample» must be identical for all methods. This also applies to the standard solution which is added in the command **ADD STD – ADD STANDARD DT**. It is not possible to have different solution names assigned to the same 807 Dosing Unit.

ADD AUX	ADD AUX	ADD AUX
ADD VMS	ADD VMS intercept	ADD Suppressor interce

 Select the solution and volume which should be added when this command is due.



- Define the volume of solution that should be added when this command is due.
- If the name of the 807 Dosing Unit differs from the specifications in 3.3.2, check on the tab «General/Hardware» that the correct 807 Dosing Unit is assigned. It has to be the one used for sampling.

LQH	LQH	LQH
DU 50 mL VMS	DU 2 mL Brightener	DU 2 mL Suppressor



If names of 807 Dosing Units differ from the specifications in 3.3.1 and 3.3.2, then check on the tab «General/Hardware» of the respective command that the correct 807 Dosing Unit is assigned.

4.1.3 Other commands

STIR	
Stirrer OFF	



Evaluation

Settings regarding evaluation and documentation of the determination are located in the «Evaluation» part of the method. The methods already include all necessary settings to determine the brightener, suppressor, or leveler concentration in an acid copper bath. However, if modifications should be necessary, here is where important parameters are found:

4.2.1 **Substances**

In the «Substances» part, settings for peak recognition and baseline parameters are defined.

4.2.2 Standards

In the «Standards» part, the concentration of the used standard solution is defined.

Please note!

If the names for standard solutions differ from the specifications in 3.4, make sure that the name of the solution in the ADD STD command (Method run) matches the name of the solution under Evaluation - Standards.

4.2.3 Calibration

In the «Calibration» part, the calibration method, such as DT, MLAT, or RC, is defined as well as the regression type of the calibration curve.

4.2.4 Results

Among other things, settings for the automatically calculated final results can be made and the database for the storage of the results can be defined in the «Results» part.

Operation

Preparation of 807 Dosing Units 5.1

For startup or shutdown of the system, the preparation of 807 Dosing Units can be done from the viva «Manual control». But more conveniently, the 807 Dosing Units can be prepared using the method «800105021EN Preparation of dosing units», which is provided with this Application

Bulletin. The method allows users to specify the number of preparation cycles individually for each 807 Dosing Unit on the workplace as shown in Figure 8.

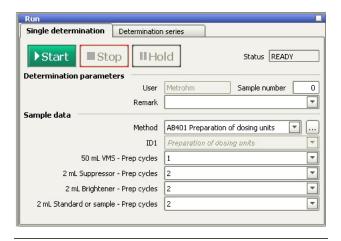


Figure 8. Example for the settings in the «Run» window to prepare the 807 Dosing Units using the method «800105021EN Preparation of dosing units».

Features of the method:

- The preparation of the 807 Dosing Unit is repeated as many times as specified on the workplace: 1 = once, 2 = twice, etc.
- When «Prep cycles» = 0, the respective 807 Dosing Unit is not prepared.
- All 807 Dosing Units with «Prep cycles» > 0 will be prepared simultaneously.

5.1.1 Manual operation



- The measuring vessel is removed, and a waste beaker is placed below the measuring head to avoid contamination of the electrodes and the measuring vessel with concentrated organic additives.
- The electrodes and the measuring vessel are rinsed with deionized water after the determination.

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5.2 Conditioning in VMS

5.2.1 Manual operation



 807 Dosing Unit «50 mL VMS» must be prepared with VMS.

5.2.2 Workplace



The sample amount is fixed at 30 mL, which is the volume of VMS required to condition the electrodes when measuring vessel 6.1415.250 is used (also see 7.5).

5.2.3 Course of events

- VMS is automatically added from 807 Dosing Unit «50 mL VMS».
- 2. The CVS sweeps are repeated until either the relative standard deviation of the Cu stripping peak is <0.5%, or for 20 runs, whatever is reached first.

5.3 Suppressor determination

5.3.1 Manual operation



- The 807 Dosing Unit «50 mL VMS» must be prepared with VMS.
- The 807 Dosing Unit «2 mL Standard or sample» has to be prepared with suppressor standard solution if a calibration is recorded, or with plating bath sample for the determination.
- The electrodes and the measuring vessel are rinsed with deionized water after the determination.

5.3.2 Workplace



- Select the sample type STANDARD to run a calibration in the «Run» window of the «Workplace». For a determination, the sample type SAMPLE has to be selected.
- No «Sample amount» needs to be defined in the «Run» window on the «Workplace» since addition volumes for the standard and the sample are defined in the method commands ADD STD and ADD SAMPLE DT.

5.3.3 Course of events

The execution of a calibration or a determination by dilution titration is controlled by the 894 Professional CVS and viva:

- VMS is automatically added from the 807 Dosing Unit «50 mL VMS».
- Either suppressor standard or plating bath sample is automatically added from the 807 Dosing Unit «2 mL Standard or sample».

5.4 Brightener determination

5.4.1 Manual operation



- The 807 Dosing Unit «50 mL VMS» must be prepared with VMS.
- The 807 Dosing Unit «2 mL Brightener» must be prepared with brightener concentrate.
- The 807 Dosing Unit «2 mL Suppressor» must be prepared with suppressor concentrate.
- The correct «Sample amount» has to be pipetted into the measuring vessel when requested.
- The electrodes and the measuring vessel are rinsed with deionized water after the determination.



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5.4.2 Workplace



- Select the sample type SAMPLE in the «Run» window of the «Workplace» to run a determination.
- Define the volume of sample used for the determination under «Sample amount» in the «Run» window of the «Workplace».

5.4.3 Course of events

The execution of the determination by modified linear approximation technique is controlled by the 894 Professional CVS and **viva**:

- The intercept solution is automatically prepared at the beginning of each determination by adding VMS from the 807 Dosing Unit «50 mL VMS» and suppressor concentrate from the 807 Dosing Unit «2 mL Suppressor».
- After the determination of the intercept value, a window will pop up prompting the user to pipette the sample on top of the intercept solution. The exact sample volume has to be pipetted manually through the pipetting opening of the 894 Professional CVS.
- 3. Standard addition is carried out automatically with brightener concentrate from the 807 Dosing Unit «2 mL Brightener».

5.5 Leveler determination

5.5.1 Manual operation



- The 807 Dosing Unit «50 mL VMS» must be prepared with VMS.
- The 807 Dosing Unit «2 mL Brightener» must be prepared with brightener concentrate.
- The 807 Dosing Unit «2 mL Suppressor» must be prepared with suppressor concentrate.
- The 807 Dosing Unit «2 mL Standard or sample» must be prepared with leveler standard.

- The correct «Sample amount» has to be pipetted into the measuring vessel when requested.
- The electrodes and the measuring vessel are rinsed with deionized water after the determination.

5.5.2 Workplace



- To run a calibration with a standard solution, select the sample type STANDARD. For a determination, the sample type SAMPLE has to be selected.
- The sample amount for sample type STANDARD has to be entered for technical reasons but has no effect on the calibration. The addition volume for the standard is defined in the method command ADD STD.

5.5.3 Course of events

The execution calibration and determination by response curve technique is controlled by the 894 Professional CVS and **viva**.

- The electrolyte solution is automatically prepared by adding VMS from the 807 Dosing Unit «50 mL VMS», suppressor concentrate from «2 mL Suppressor», and brightener concentrate from «2 mL Brightener».
- 2. After determination of the electrolyte value, leveler standard or plating bath sample are added.
 - a. In the case of calibration, the volume of leveler standard defined in the command ADD STD in the method is added from the 807 Dosing Unit «2 mL Standard or sample».
 - b. In the case of sample determination, the volume of plating bath sample as specified under sample amount has to be added manually:
 - I. In the method «with dilution», the sample has to be pipetted on top of the electrolyte solution
 - II. In the method «no dilution», the measuring vessel has to be emptied before the sample can be pipetted.
- 3. Suppressor concentrate from the 807 Dosing Unit «2 mL Suppressor» and brightener concentrate from the 807 Dosing Unit «2 mL Brightener» are automatically added after the sample.



- In the method «with dilution», the volumes for suppressor and brightener concentrate differ from the volumes used for the electrolyte solution.
- b. In the method «no dilution», the volumes for suppressor and brightener concentrate are similar to the volumes used for the electrolyte solution.
- c. In the case of calibration, no additional suppressor or brightener is added.
- 4. The software calculates the regression data for the calibration or the concentration of leveler in the plating bath sample.

6 Additional 807 Dosing Units

With the hardware as specified in chapter 1, the number of solutions which are immediately available for applications is restricted to four. However, the setup can be extended beyond the four 800 Dosinos shown in **Figure 3**. This requires additional hardware and some additional settings in the software.

6.1 Instruments and accessories

- 800 Dosinos are not part of the equipment supplied with 807 Dosing Units (6.5339.500 or 6.5339.520), which is why two 800 Dosinos have to be added to each.
- If more than four 800 Dosinos should be connected, then another 846 Dosing Interface is required for every four additional 800 Dosinos.
- If more than four 807 Dosing Units with 2 mL should be connected, then an additional four-way micro dosing tip (6.1824.000) and a ball stopper (6.1446.030) are required.
- If more than four 807 Dosing Units should be connected to the waste bottle, then an additional three-way stopper (6.1543.210) is needed.

The following two tables show examples for instruments and accessories which are required when the system listed in chapter 1 should be extended by equipment with two 807 Dosing Units (2 mL + 2 mL, or 2 mL + 50 mL, respectively).

6.1.1 6.5339.500

Accessories required when extending the system described in chapter 1 with equipment 6.5339.500 (two 807 Dosing Units 2 mL + 2 mL):

Quantity	Article no.	Designation
2	2.800.0020	800 Dosino
1	2.846.0010	846 Dosing Interface
1	6.5339.500	Equipment two 807 Dos- ing Units (2 mL + 2 mL) for VA/CVS
1	6.1824.000	Four-way micro dosing tip
1	6.1446.030	Ball stopper
1	6.1543.210	3-way stopper

6.1.2 6.5339.520

Accessories required when extending the system described in chapter 1 with equipment 6.5339.520 (two 807 Dosing Units 50 mL + 2 mL):

Quantity	Article no.	Designation
2	2.800.0020	800 Dosino
1	2.846.0010	846 Dosing Interface
1	6.5339.520	Equipment two 807 Dosing Units (50 mL + 2 mL) for CVS
1	6.1543.210	3-way stopper

6.2 Electrical connections

A maximum of four 800 Dosinos can be connected directly to the 894 Professional CVS. For electrical connection of additional 800 Dosinos, an 846 Dosing Interface is required. Up to four 800 Dosinos can be connected to an 846 Dosing Interface.

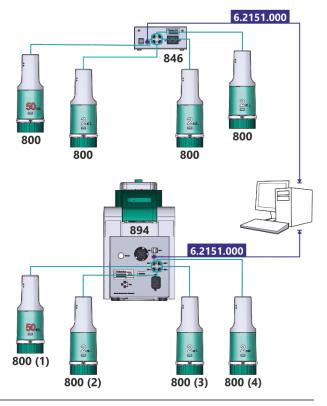


Figure 9. Connecting more than four 800 Dosinos using the 846 Dosing Interface.

6.3 Tubing connections

For the general setup of 2 mL and 50 mL dosing units, see chapters 2.2.2 and 2.2.3. The following chapters describe how to connect additional solutions to the 894 Professional CVS.

6.3.1 Volumes < 2 mL

Volumes of <2 mL are usually added via the four-way micro dosing tip 6.1824.000. If more than four small volumes should be added, an additional four-way dosing tip is required. It can be installed in the measuring head on the left side together with a ball stopper 6.1446.030.

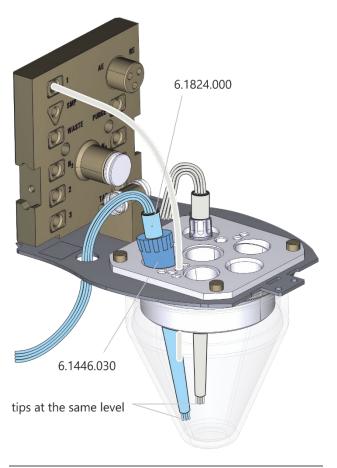


Figure 10. Additional four-way micro dosing tip installed in the measuring head.

Please note!

- A maximum of eight solutions can be added via capillaries with this setup.
- Since this setup uses the left SGJ 14 opening, the temperature sensor 6.1110.120 cannot be installed in the measuring head at the same time.



6.3.2 Volumes > 2 mL

For the addition of volumes >2 mL, a tubing with a larger inner diameter should be used. The 2 mm FEP tubing 6.1805.120 coming from an additional 50 mL dosing unit can be connected to connector 2 on the back of the 894 Professional CVS. In addition, a tubing with short bend protection from the set 6.1829.070 has to be installed between connector 2 of the adapter plate of the measuring head and the measuring head insert.

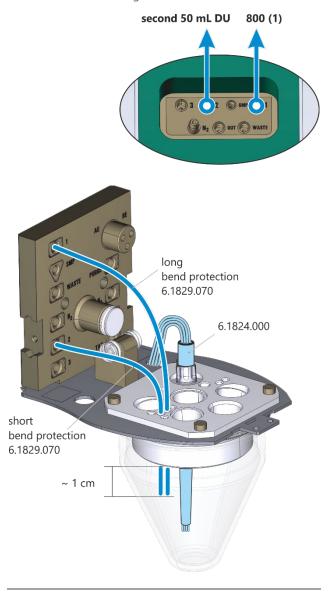


Figure 11. Tubings for two 807 Dosing Unit with 50 mL.

6.3.3 Waste

Port 3 of an 807 Dosing Unit is connected to a waste bottle via a 2 mm FEP tubing (6.1805.530) to speed up the preparation and to minimize contaminating the measuring vessel.

If more than four 807 Dosing Units should be connected to the waste bottle, then a 3-way stopper 6.1543.210 can be installed in the SGJ opening of the bottle head. The antidiffusion valves are removed.

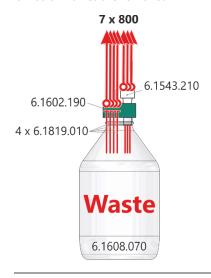


Figure 12. Configuration for more than four tubings on the waste bottle.

Please note!

- This setup can be used for a maximum of seven waste tubings.
- The waste bottle must not be closed completely. The central 3 mm hole of the bottle head must be left open for pressure balancing. Overpressure can damage the 800 Dosinos.
- Larger volumes from 50 mL dosing units are added via 2 mm FEP tubing, which is why preparation of these dosing units can also be done via Port 1 of the 807 Dosing Unit. In this case, the setting for «Dosing port Prep/Empty» in the viva «Configuration» would need to be changed to «Dosing port 1».



6.4 Configuration and viva «Method»

6.4.1 Procedure

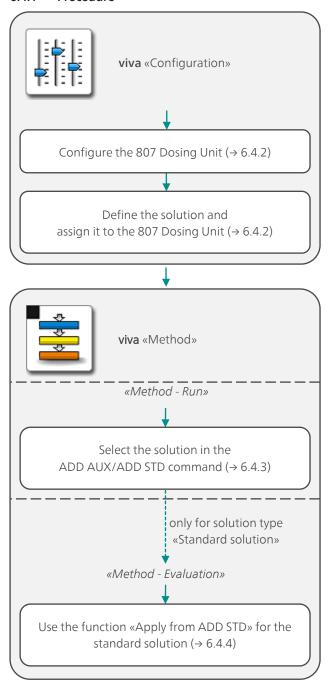


Figure 13. Recommended order of steps for the definition of new solutions for automatic addition.

6.4.2 Define 807 Dosing Unit and solution

To make solutions in **viva** available for automatic addition, the solution first needs to be specified in the configuration and must be assigned to an 807 Dosing Unit. For the configuration of 807 Dosing Units and definition of solutions, see chapters 3.3 and 3.4.

6.4.3 Use the solution in the method run

To use a solution in a method, select it from the drop-down list in the corresponding **ADD AUX** or **ADD STD** command. Make sure the option «Addition – Add with dosing device» is selected.

6.4.4 Adapt the solution name for the standard solution in the evaluation part

For a correct assignment of the standard volume and the concentration of the analyte in the standard solution, the spelling of the name of the solution used in an **ADD STD** command in the method run must be identical to the name in the table of standard solutions in the evaluation subwindow (EVALUATION > STANDARDS).

To ensure this, the following procedure should be used:

- 1. Delete the column with the current standard.
- Add a new standard by using the function «Apply from ADD STD» which can be either accessed with a right mouse click on an empty column or via the «Edit ▼» button.



3. Adapt the concentration information to the concentration of the analyte used in the standard solution of the application.



7.1 884 Professional VA

Instead of the 894 Professional CVS (2.894.1210) listed in chapter 1, the 884 Professional VA (2.884.1210) can be also used in the setup, as well as the two instrument versions with three 800 Dosinos (2.894.1310, 2.884.1310).

7.2 Storing 807 Dosing Units

When an 807 Dosing Unit is not used for a longer period of time (e.g., during the night, over the weekend), it should be rinsed with deionized water. For storage, the 807 Dosing Unit should not be emptied. Instead, it is kept with the dosing cylinder filled with deionized water.

When highly concentrated solutions, such as VMS, additive concentrates, or plating bath samples, are left in the 807 Dosing Unit, crystals or elemental copper can form. This can damage the 807 Dosing Unit in various ways. Deposits between the valve and distributor disk can cause them to stick them together or form leakages. Crystals in the dosing cylinder can compromise the sealing between the dosing piston and the glass cylinder. Crystals in a capillary tubing can cause blockages which can ultimately damage the 800 Dosino if the overpressure causes any solution to leak into the electronics.

Please also refer to the 807 Dosing Unit manual for recommendations regarding maintenance and greasing of 807 Dosing Units.

7.3 Storing electrodes

When the electrodes are not used (e.g., during the night, over the weekend), then they should be thoroughly rinsed.

Working and auxiliary electrodes can either be stored in deionized water or dry.

The reference electrode should be stored separately in a storage vessel (e.g., 6.2008.040). The vessel can either be filled with KCl solution (c(KCl) = 3 mol/L), or KNO $_3$ solution (c(KNO $_3$) = 1 mol/L) in case KNO $_3$ is used as bridge electrolyte. Filling the storage vessel to the maximum level prevents the reference electrode from running dry.

7.4 Waste bottle

The waste bottle must not be closed completely. For pressure balancing in the waste bottle, keep at least one opening unsealed. Overpressure can lead to a malfunction of the 800 Dosinos.

7.5 Measuring vessel

With the electrode equipment for CVS (e.g., 6.5339.010), two different measuring vessels with different capacities are supplied (see **Table 2**). The minimum volume is given by the fact that all three electrodes must be immersed into solution. The limit for the maximum volume results from the distance between the solution level and the bottom of the measuring head insert, which should be at least 1 cm. The measuring vessel has to be selected according to the volumes used in the applications. To fit into the limits of the measuring vessel, volumes in the application can also be adapted.

Table 2. Capacities of measuring vessels.

Measuring vessel	Min. volume	Max. volume
6.1415.210	10 mL	70 mL
6.1415.250	30 mL	150 mL

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