

Agilent 850-DS Sampling Station Volume Calibration

Volume calibration procedure

This document details procedures for calibration and verification of volume accuracy for the Agilent 850-DS Sampling Station for systems configured with either USP Apparatus 1/2 or 3/7.

1. Add an appropriate volume of purified water to each active dissolution vessel.
2. Allow the temperature of each vessel to equilibrate to 37.0 ± 0.5 °C.
Note: Other temperatures can be used (e.g., 32.0 ± 0.5 °C), which are appropriate to the dissolution tests. Ensure that the density value is appropriate in the calculations for Step 14.
3. Ensure each sampling cannula of the dissolution apparatus is completely submerged in the water of the dissolution vessel(s).
4. Label an appropriate number of test tubes based on the 850-DS being qualified and record their tare weight in the table beneath Step 14.
5. Place the weighed and labelled test tubes in row 1 (front row) of the 850-DS sample tray.
6. Place an additional set of test tubes (empty, not weighed) into row 2 of the 850-DS sample tray.
7. From the 850-DS main screen press **Calibration** on the 850-DS keyboard.
8. Complete the following table:

Action	Volume (mL)
mL to Dispense	10.0
Row Number	2
Actual mL	N/A (leave empty)
mL/Stroke	N/A

- Press **Dispense**. The 850-DS pumps 10 mL and dispenses the volume into row 2 of the sample tray. These test tubes need not be weighed. This step was performed only to prime the sample lines.
- Adjust the Row Number from 2 to 1.
- Press **Dispense**. The 850-DS pumps 10 mL and dispenses the volume into row 1 of the sample tray.
- Record the gross weight of each sample plus the test tube in the table beneath Step 14.
- Calculate the net weight of each sample by subtracting the tare weight from the gross weight and record the value in the table beneath Step 14.
- Calculate the volume of each sample using the following formula, and record it in the following table:

$$\text{Sample Volume (mL)} = \frac{\text{Sample Net Wt. (g)}}{0.993 \text{ g/mL}}$$

Where 0.993 g/mL is the density of water at 37.0 °C (extrapolated from the Density of Water table found in the *CRC Handbook of Chemistry and Physics*). This value is indicative of the temperature of the water at the time of sampling. If other temperatures are used, then correct the density accordingly.

Temperature (°C)						
Density (g/mL)						
Vessel Position	Tube/Vial Tare Wt. (g)	Tube/Vial Gross Wt. (g)	Sample Net Wt. (g)	Sample Volume (mL)	N/A	
1						
2						
3						
4						
5						
6						
7						
8						
Mean Sample Volume (mL)						

- Calculate the mean sample volume (mL) dispensed for all eight test tubes and record it in the table above. Enter this value in the "Actual mL" field on the 850-DS. The 850-DS will calculate a new calibration volume that appears in the "mL/stroke" field.

Steps 1 to 15 were completed successfully.	(Indicate pass/fail/NA)
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Volume accuracy check – apparatus 1/2

- Place clean, dry test tubes in all positions of the sample tray.
- Place the dissolution vessel or alternate container (e.g. graduated cylinder) on an appropriate analytical balance and tare the balance.
- Fill the vessel or container with approximately 900 mL of purified water and record the initial weight in the table beneath Step 28.
- Repeat Steps 2 to 3 for all applicable vessel positions.
- If your system configuration includes a 705-DS, or a non-Agilent apparatus, place each resident probe into the corresponding dissolution vessel.
- From the 850-DS main screen, select the Methods tab. Press the number of any location with no stored method.
- Enter the following dissolution test parameters in the fields on the 850-DS screen:

Option	Response
Name	VolCheck
Vessel Temp (°C)	37.0*
Bath Temp (°C)	37.2*
Spindle Speed (RPM)	50
Media Volume (mL)	900
Rotation Start Delay (s)	3
Apparatus	2
Enable Manifold	Initial Temp
	Final Temp
Profile Interval (minutes)	5:00
Enable Final Spin	Checked
Final Spin Speed (RPM)	250.0
Final Spin Durham (minutes)	1:00

* Use the appropriate temperature from the previous volume calibration.

- Press **NEXT**.
- Select each option and enter the response as listed in the following table:

Option	Response
Sample Volume (mL)	10.0
Prime Volume (mL)	5.0
Purge Volume (mL)	5.0
Waste Drop Volume (mL)	0.5
Media Change	Disabled
Enable Media Replacement	Checked
	10.5 mL
Enable Cleaning Cycle	Unchecked

- Press **NEXT**.

11. Enter the following time points in the table on the 850-DS screen:

Option	Response
Sample Timepoints	Row 1: Enter 000:06:00
	Row 2: Enter 000:12:00
	Row 3: Enter 000:18:00
	Row 4: Enter 000:24:00
	Row 5: Enter 000:30:00
	Row 6: Enter 000:36:00
	Row 7: Enter 000:42:00
	Row 8: Enter 000:48:00
	Row 9: Enter 000:54:00
	Row 10: Enter 001:00:00
	Row 11: Enter 001:06:00
*Infinity spin time point will be collected in Row 12	

12. Press **SAVE** to save the method and return to the 850-DS main screen.
13. Ensure the water bath, if applicable, and vessels are up to temperature before running the program.
14. Properly label and record the tare weight in grams of the test tubes for rows 1, 6, and 12 in the appropriate table beneath Step 23.
15. From the 850-DS main screen, press **RUN**.
16. Select the method corresponding to the OQ TEST and enter the following information on the Start Options screen:

Operator	(Your initials)
Lot	ABC
Batch	123
Note	QC
Start Options	Instant

17. Ensure that Dissolution Apparatus Control is checked if connected to an Agilent dissolution apparatus.
18. Press **RUN**.
19. Attach a printout of the 850-DS report to this guide.
20. Visually inspect all sample tubes to ensure the volume levels are consistent.
21. Record the gross weight (in grams) of the test tubes and samples for rows 1, 6, and 12 in the appropriate table beneath Step 23.

22. Calculate the net weight of each sample by subtracting the tare weight from the gross weight and record the value (in grams) in the appropriate table beneath.
23. Calculate the volume of each sample using the following formula and record it in the appropriate table:

$$\text{Sample Volume (mL)} = \frac{\text{Sample Net Wt. (g)}}{0.993 \text{ g/mL}}$$

Where 0.993 g/mL* is the density of water at 37.0 °C (extrapolated from the Density of Water table found in the *CRC Handbook of Chemistry and Physics*). This value is indicative of the temperature of the water at the time of sampling. If other temperatures are used, then correct the density accordingly.

Temperature (°C)					
Density (g/mL)					
Table 1, Row 1					
Vessel Position	Tube/Vial Tare Wt. (g)	Tube/Vial Gross Wt. (g)	Sample Net Wt. (g)	Sample Volume(mL)	N/A
1					
2					
3					
4					
5					
6					
7					

Table 2, Row 6					
Vessel Position	Tube/Vial Tare Wt. (g)	Tube/Vial Gross Wt. (g)	Sample Net Wt. (g)	Sample Volume(mL)	N/A
1					
2					
3					
4					
5					
6					
7					
8					

Table 3, Row 12					
Vessel Position	Tube / Vial Tare Wt. (g)	Tube / Vial Gross Wt. (g)	Sample Net Wt. (g)	Sample Volume (mL)	N/A
1					
2					
3					
4					
5					
6					
7					

24. Record the results of the 850-DS Accuracy Check procedure for Apparatus 1 and 2 in the following table:

Condition	Pass	Fail	N/A
Each Individual Sample Volume for Rows 1, 6, and 12 Meets the Applicable Acceptance Criteria Listed Below.			
Acceptance Criteria: Set volume = Sample volume ± 0.2 mL			
Steps 1 to 24 Were Completed Successfully.	(Indicate pass/fail/NA)		

Note: A similar procedure should be followed if connected to an Agilent USP 3 BIO-DIS II Dissolution Apparatus. Simply fill the outer tubes with water and ensure that the sampling cannulas are fully submerged. Create a sample method with 12 timepoints to fill each test tube and execute Steps 12 to 24 above.

www.agilent.com/chem/dissolution

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This information is subject to change without notice.

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