



DISSOLUTION SYSTEMS

OPERATIONAL QUALIFICATION

Standard OQ Test Suite

This document describes the test program for qualifying dissolution instruments, and the following tables list all OQ tests.

Apparatus 1, 2, 5, and 6

Test	Setpoints and Parameters	Limits
Wobble	App 1 measured at basket rim; app 2 at paddle shaft; rotation speed of 50 RPM for all measurements	Measurement is ± 1.0 mm from the axis
Shaft Verticality	Measurement 1: point on vertical axis Measurement 2: 90° from measurement 1	Measurements are $\leq 0.5^\circ$ from vertical*
Vessel Centering	Center position set; lower point is measured	Measurement is ≤ 2.0 mm from centerline
Rotational Speed (RPM)	Speed 1: 50 RPM (for all positions) Speed 2: 100 RPM (for one position only) Speed 2: 250 RPM (for one position only)	Speeds are $\pm 4.0\%$ RPM from setpoint
Vessel Temperature Accuracy	Temperature: 37°C	Accuracy $\leq 0.5^\circ\text{C}$
Vessel Temperature Stability	Temperature: 37°C	Stability $\leq 0.5^\circ\text{C}$

* Or within bubble if a mechanical (vs. digital) gauge is used

Apparatus 3 and 7 / BioDis

Test	Setpoints and Parameters	Limits
Vessel Temperature Accuracy	Temperature: 37°C (App3) Temperature: 32°C (App7)	Accuracy $\leq 0.5^\circ\text{C}$
Manual Hold Dip Verification	Dip time setpoint: 10 seconds	Time is setpoint ± 1 second
Reciprocation Distance Verification	Stroke length: 100 mm (App 3) Stroke length: 20 mm (App 7)	Accuracy: ≤ 1 mm (App 3) Accuracy: ≤ 2 mm (App 7)
Dips Per Minute (DPM)/ Reciprocation Frequency Verification	Setpoints 1, 2, and 3: 5, 15, and 30 DPM	Accuracy: $\leq 5.0\%$

Apparatus 7 / 400DS

Test	Setpoints and Parameters	Limits
Vessel Temperature Accuracy	Temperature: 37°C	Accuracy $\leq 0.5^\circ\text{C}$
Reciprocation Distance Verification	Stroke length: 20 mm	Accuracy: ≤ 1 mm
Dips Per Minute (DPM)/ Reciprocation Frequency Verification	Setpoints 1, 2, and 3: 5, 15, and 30 DPM	Accuracy: $\leq 5.0\%$
Volume Accuracy (applies only if an autosampler/injector is installed)	Tests each individual sample volume for rows 1, 5, 9	0.99 – 1.01 ml (1.5 ml vials) 2.97 – 3.03 ml (4.0 ml vials)

850DS Autosamplers

Test	Setpoints and Parameters	Limits
Volume Accuracy	Sample volume: 10.0 ml Tests each individual sample volume in rows 1, 6, 11	Accuracy ± 0.25 ml
Media Replacement Accuracy	Volume: 900 ml	Volume difference ≤ 9 ml
Clock and Alarm Verification	Injector clock set to 00:00:00 Alarm set to sound after 1 minute	After an hour, clock shows 01:00.00 ± 5 seconds Alarm sounds after 1 minute

VK8000 Autosamplers

Test	Setpoints and Parameters	Limits
Volume Accuracy	Sample volume: 10.0 ml Tests each individual sample volume in rows 0, 6, 9	Accuracy ± 0.5 ml (peristaltic pump) Accuracy ± 0.2 ml (syringe pump) Accuracy ± 0.3 ml (syringe pump plus filter changer)
Media Replacement Accuracy	Sample volume: 900 ml	Volume difference ≤ 10 ml
Clock and Alarm Verification	Injector clock set to 00:00:00 Alarm set to sound after 1 minute	After an hour, clock shows 01:00.00 ± 5 seconds Alarm sounds after 1 minute

VK8020 Autosampler

Test	Setpoints and Parameters	Limits
Volume Accuracy	Sample volume: 10.0 ml Tests each individual sample volume for rows 0, 6, 9	Accuracy ± 0.5 ml
Syringe Pickup Volume Verification	Vials 1 and 2 tested for 25% and 99% withdrawals	Accuracy* $\leq 5\%$ theoretical volume
Clock Alarm Verification	Injector clock set to 00:00:00 Alarm set to sound after 1 minute	After an hour, clock shows 01:00.00 ± 5 seconds Alarm sounds after 1 minute

* % nominal volume (accuracy between actual volume and theoretical volumes)

Test Design and Rationale

Wobble

Description: This test evaluates wobble which is calculated as the absolute value of the difference between the maximum and minimum readings.

Procedure (Shaft for App 2): A runout gauge is placed on top of the vessel plate, and the drive module is positioned so that the gauge probe touches the shaft about 2 cm above the top of the paddle blade or basket. The gauge is placed so that the probe slightly presses in on the turning shaft. If a mechanical gauge is used, the gauge's pointer should read slightly more than zero. The pointer will vary from a minimum to a maximum reading, and the difference is called the wobble.

Procedure (Basket for App 1): A runout gauge is placed on top of the vessel plate and the drive unit is positioned so that the gauge probe touches the bottom rim of the basket. The gauge is placed so that the probe slightly presses in on the turning shaft. If a mechanical gauge is used, the gauge's pointer should read slightly more than zero. The pointer will vary from a minimum to a maximum reading and the difference is called the wobble.

Shaft Verticality

Description: This test verifies shaft verticality in two directions, 90° apart on the vertical axis.

Procedure: Lower the drive unit to where it would be during an actual dissolution test. If necessary the shaft verticality may be checked with the shafts raised above the drive unit. Place an accurate bubble level on the front edge of each of the shafts. The bubble should be within the lines of the level. Rotate the level 90° so it is on the side of the shaft. The bubble should again be within the lines of the level for each shaft. If the shafts are not vertical adjust the feet of the apparatus until they are vertical. A digital leveling device may also be used to determine the shaft verticality. The shaft must be $\leq 0.5^\circ$ from vertical.

Vessel Centering

Description: This test verifies vessel centering.

Procedure: A mechanical or digital centering device that centers the inside of the vessel around the shaft (or surrogate shaft) can be used. The centering is measured in the vessel's cylindrical portion just above the bottom portion of the vessel. The shaft (or surrogate shaft) must be centered within 2.0 mm from the center line.

Rotational Speed (RPM)

Description: This test verifies rotational speed of the shafts.

Procedure: A tachometer is used to measure the rotational speed of the paddle or basket. The shafts should be rotating smoothly at ± 4 rpm of the target value.

Vessel Temperature Accuracy

Description: This test uses a calibrated thermometer to verify that the media temperature reaches the system setpoint.

Procedure: This test uses a calibrated thermometer to verify that the media temperature reaches the system setpoint.

Vessel Temperature Stability

Description: This test uses a calibrated thermometer to determine the stability of the vessel temperature.

Procedure: This test uses a calibrated thermometer to determine the stability of the vessel temperature. Stability is calculated as the delta between the highest and lowest measured temperatures.

Manual Hold Dip Verification

Description: This test verifies that sample cell or holder is held at the bottom of the stroke for the specified duration.

Procedure: This test uses a calibrated timer to verify that the dipping is paused for a set period of time before starting a run.

Reciprocation Distance Verification

Description: This test verifies the length of the dip stroke.

Procedure: This test uses a calibrated ruler to verify the length of the dip stroke.

Dips Per Minute (DPM)/ Reciprocation Frequency Verification

Description: This test verifies that the system performs the programmed number of dips per minute.

Procedure: This test uses a calibrated timer to verify the programmed number of dips per minute.

Volume Accuracy

Description: This test verifies the accuracy of the withdrawn sample volume.

Procedure: This test verifies accuracy using the weight and density of the water withdrawn.

Media Replacement Accuracy

Description: This test verifies that the amount of sample withdrawn is accurately replaced.

Procedure: This test verifies that the sampled volume is replaced accurately by using the initial/final weights and density of the water in the vessel.

Syringe Pickup Volume Verification

Description: This test verifies that the system withdraws the programmed volume.

Procedure: This test is completed only if an autosampler is configured in the system.

Clock Alarm Verification

Description: This test verifies that the injector clock and alarm are working properly.

Procedure: The injector clock is set to 00:00:00 and should be within 5 seconds of 1 hour after 60 minutes elapse on the calibrated timer. The alarm is set to sound after one minute.

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