Designation: E2503 – 13 (Reapproved 2020)

Standard Practice for Qualification of Basket and Paddle Dissolution Apparatus[1]

This standard is issued under the fixed designation E2503; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the set-up and calibration of the paddle and basket dissolution apparatus.

1.2 Use of this practice may be applied to apparatus that have been modified to enable automatic dissolution testing (that is, a valve in the bottom of the vessel or sampling through the shaft).

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Significance and Use

2.1 This practice outlines a procedure for the mechanical calibration of paddle and basket dissolution units to ensure reproducibility of results.

2.2 Once a unit meets all of the mechanical specifications included in this practice, it is considered calibrated and further calibration with dissolution calibrator tablets is not required.

3. Analyst Responsibilities

3.1 Verify the vessel, basket, and paddle dimensions on receipt through measurement or Certificate of Analysis (COA) or Certificate of Conformity (COC).

3.2 Ensure the instrument is calibrated and fit for performing dissolution analysis.

4. Procedure

4.1 *Background*—The set-up, mechanical, and operational checks are used to minimize variability during dissolution testing to ensure the reproducibility of dissolution results.

4.2 Wherever possible, tools shall be traceable to an accepted standard calibration source from a national or international calibration laboratory.

4.3 *Apparatus Set-up*—During apparatus installation or after replacement of parts or components, verify that the description and critical dimensions for each part meets the original description and dimension.

4.3.1 *Vessel Dimensions*—In the absence of a COA or COC, the vessel's internal dimensions should be measured with an appropriate measuring device and vessel shape and condition should be noted. For example, for a cylindrical, hemispherical vessel, the vessel's sides must be cylindrical, the internal dimension should be measured, and the vessel bottom should be smooth and without defects. The vessel must fit within the apparatus in such a manner as to ensure stable operation and centering of the shaft in the vessel.

4.3.2 *Basket/Shaft Dimensions*—In the absence of a COA or COC, an appropriate measuring device is used to measure the relevant basket dimensions. Critical dimensions to be measured on each basket should include but are not limited to: shaft diameter, vent hole diameter, thickness of wide portion of the basket-to-shaft adaptor, total basket height, internal diameter at the top of the basket, outer diameter of the screen, height of the open screen, outer diameter of bottom, diameter of screen on the bottom, and screen mesh number.

4.3.3 *Paddle Dimensions*—In the absence of a COA or COC, an appropriate measuring device is used to measure the relevant dimensions of the paddle. Examples of dimensions to be determined on each paddle should include but are not limited to: shaft diameter, blade height, blade thickness, total blade length, length of flat portion on bottom of blade, radius of the angle on the top outer edge of the top of the blade, radius of the outside edge of the blade, difference between the distance from the midline of the shaft to the top outer edge for the two sides, and difference between the heights of both sides of the paddles at the outside top.

4.4 *Maintenance*—Consult the manufacturer's maintenance recommendations and maintenance schedule to establish an

[1] This practice is under the jurisdiction of ASTM Committee E55 on Manufacture of Pharmaceutical and Biopharmaceutical Products and is the direct responsibility of Subcommittee E55.03 on General Pharmaceutical Standards.

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appropriate maintenance program based on the frequency of apparatus use and quality system requirements.

4.5 *Mechanical Calibration*—Perform these tests on the frequency determined by the quality system or after repair or move. If the instrument is not in routine use, the mechanical calibration may be performed before performing the first dissolution test. Some instrument manufacturers supply special tools or incorporate automatic mechanical calibration devices within their equipment, and these may be used. Depending on the adjustments necessary to meet the mechanical calibration criteria, the position and orientation of each vessel may need to be noted to ensure proper subsequent set-up.

4.5.1 *Shaft Wobble*—A runout gauge is positioned so that the gauge probe touches the turning 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. The absolute value of the difference between the maximum and minimum readings is the wobble. The measured value must not exceed 1.0 mm total runout.

4.5.2 *Paddle and Basket Shaft Verticality*—Use an accurate bubble level or digital leveling device to determine that the shafts are vertical in two directions 90° apart around the vertical axis while the drive unit is in the operating position. If a bubble level is used, the bubble should be centered within the lines of the level. If necessary, the verticality may be checked with the shafts raised above the drive unit.

4.5.3 *Basket Wobble*—A runout gauge is positioned so that the gauge probe touches the bottom rim of the turning basket. The absolute value of the difference between the maximum and minimum readings is the wobble. The measured value must not exceed 1.0 mm total runout.

4.5.4 *Vessel Centering*—A mechanical or digital centering device that measures centering inside the vessel is required. The centering device is used to center the vessels around the paddle or basket shafts or a surrogate shaft at two different positions: at the top rim of the vessel and above the bottom portion of the vessel just above the basket or paddle position. Slowly turn the shaft and check the centering at both levels. If the vessel is not centered on either level, adjust the vessel per manufacturer's recommendations to make it centered. Repeat this process until both bottom and top positions are centered within 1 mm from the center line.

4.5.5 *Vessel Verticality*—After raising the apparatus head, a leveling device can then be placed on the inside wall of the vessel. Measure the vessel at two points, 90° apart. The vessel must not be more than 1.0° from vertical at either position. Alternatively, calculations can be done to determine vessel verticality using information from a centering device (two measurements taken directly above each other at a known height apart for each calculation). Verticality should be calculated at two places 90° apart.

4.5.6 *Basket and Paddle Depth*—If the height of the paddle/basket is adjustable, a depth gauge is used to set the distance between the bottom of each of the paddle blades or baskets and the bottom of the vessels. An adjustable depth gauge or other device can be used to verify that the paddle/basket height is within 8 % of desired height. For example, for a paddle at 25 mm from the bottom, the height needs to be ±2 mm.

4.5.7 *Rotational Speed*—A tachometer is used to measure the rotational speed of the paddle or basket. The rate of rotation must be within 2 % or ±2 rpm of the stated rate, whichever is larger.

4.5.8 *Vessel's Temperature Sensor*—If the system has a thermal sensor in each vessel to check the temperature of the medium, the performance of each thermal sensor should be verified against a traceable standard.

4.6 *Operation*—Before each dissolution test perform the following:

4.6.1 *Vessel Examination*—Each vessel must be free of scratches, cracks, pits, and residue.

4.6.2 *Basket Examination*—Each basket must be free of defects such as rusting or corrosion, any wires sticking out beyond the basket, clogged mesh holes, and dented mesh sides or bottom. Make sure the basket is not deformed from its original configuration. Verify that the method of attaching the basket to the shaft (that is, clips, O-rings, and so forth) is the one described in the analytical method to be used.

4.6.3 *Paddle Examination*—Each paddle must be visually examined for defects such as rusting and loose pieces of coating sticking out from the paddles (for paddles coated with polytetrafluoroethylene (PTFE) or another coating).

4.6.4 *Vessel Temperature*—The temperature of the medium inside each vessel is measured at time of use. The medium temperature must be ±0.5°C from the target value.

4.6.5 *Vibration*—There can be no significant vibration in the dissolution apparatus or medium. Possible sources of vibration are the surrounding environment, the dissolution unit itself or one of its components or an external water bath circulating heater.

5. Report

5.1 The date, analyst, and the dissolution apparatus's manufacturer, model number, and serial number should be recorded on an appropriate Mechanical Calibration Report Sheet along with the appropriate observations. Calibration reports need to include the results obtained originally and the results after any adjustments or replacements are made. Examples of report forms for apparatus that use six 1-L vessels that are cylindrical with a hemispherical bottom, 165 mm high and 100 mm in diameter with paddle or basket 25 mm from the bottom are found in Appendix X1 and Appendix X2.

6. Keywords

6.1 basket dissolution apparatus; calibration; paddle dissolution apparatus

APPENDIXES

(Nonmandatory Information)

X1. BASKET APPARATUS QUALIFICATION REPORT SHEET

Date _____ Analyst _____

Dissolution Apparatus: Manufacturer _____ Model #_____ Serial #_____ Dissolution Vessels: Manufacturer _____

MECHANICAL CALIBRATION REPORT SHEET—BASKET APPARATUS

Calibration Parameter	Point of Measurement	Results & Comments	Tools Used	Specifications
Shaft wobble	2 cm above top of basket	1. _____ 2. _____ 3. _____ 4. _____ 5. _____ 6. _____		≤1.0 mm total runout
Shaft verticality	Along shaft	Record results at 2 points that are 90° apart. Shaft is vertical: (Y/N) Shaft1 Pt1: _____ Pt2: _____ Shaft2 Pt1: _____ Pt2: _____ Shaft3 Pt1: _____ Pt2: _____ Shaft4 Pt1: _____ Pt2: _____ Shaft5 Pt1: _____ Pt2: _____ Shaft6 Pt1: _____ Pt2: _____		Shaft must be vertical. The bubble should be centered within the lines of the level.
Basket wobble	Bottom of basket rim	1. _____ 2. _____ 3. 4. 5. _____ 6. _____		≤1.0 mm total runout
Vessel/Shaft centering	Step 1: Measured lower position Step 2: Measured upper position Note: Measurements are in the straight portion of the vessel.	1. _____ Step 1: 2. _____ 3. _____ 4. _____ 5. _____ 6. _____ 1. _____ Step 2: 2. _____ 3. _____ 4. _____ 5. _____ 6. _____		Within 1 mm from centerline
Vessel verticality	Straight portion of vessel at two places 90°apart	1-1. _____ 2. _____ 2-1. _____ 2. _____ 3-1. _____ 2. _____ 4-1. _____ 2. _____ 5-1. _____ 2. _____ 6-1. _____ 2. _____		≤1.0° from vertical
Height check/Basket depth	Basket Bottom	1. _____ 2. _____ 3. _____ 4. _____ 5. _____ 6. _____		±8 % or 25 ± 2 mm
Rotational speed		50 rpm _____ 100 rpm _____		Larger of ±2 % or ±2 rpm
Vessel temperature sensor		1. _____ 2. _____ 3. _____ 4. _____ 5. 6.		±0.5 % from target value

X2. PADDLE APPARATUS QUALIFICATION REPORT SHEET

Date _____ Analyst _____

Dissolution Apparatus: Manufacturer _____ Model #_____ Serial #_____ Dissolution Vessels: Manufacturer _____

MECHANICAL CALIBRATION REPORT SHEET—PADDLE APPARATUS

Calibration Parameter	Point of Measurement	Results & Comments	Tools Used	Specifications
Shaft wobble	2 cm above top of paddle	1. _____ 2. _____ 3. _____ 4. _____ 5. _____ 6. _____		≤1.0 mm total runout
Shaft verticality	Along shaft	Record results at 2 points that are 90° apart. Shaft is vertical: (Y/N) Shaft1 Pt1: _____ Pt2: _____ Shaft2 Pt1: _____ Pt2: _____ Shaft3 Pt1: _____ Pt2: _____ Shaft4 Pt1: _____ Pt2: _____ Shaft5 Pt1: _____ Pt2: _____ Shaft6 Pt1: _____ Pt2: _____		Shaft must be vertical. The bubble should be centered within the lines of the level.
Vessel/Shaft centering	Step 1: Measured lower position Step 2: Measured upper position Note: Measurements are in the straight portion of the vessel.	Step 1: 1. _____ 2. _____ 3. _____ 4. _____ 5. _____ 6. _____ Step 2: 1. _____ 2. _____ 3. _____ 4. _____ 5. _____ 6. _____		Within 1 mm from centerline
Vessel verticality	Straight portion of vessel at two places 90°apart	1-1. _____ 2. _____ 2-1. _____ 2. _____ 3-1. _____ 2. _____ 4-1. _____ 2. _____ 5-1. _____ 2. _____ 6-1. _____ 2. _____		≤1.0° from vertical
Height check/Paddle depth	Paddle Bottom	1. _____ 2. _____ 3. _____ 4. _____ 5. _____ 6. _____		±8 % or 25 ± 2 mm
Rotational speed		50 rpm _____ 100 rpm _____		Larger of ±2 % or ±2 rpm
Vessel temperature sensor		1. _____ 2. _____ 3. _____ 4. _____ 5. _____ 6. _____		±0.5 % from target value