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USP DISSOLUTION CALIBRATOR, NON-DISINTEGRATING TYPE

Salicylic Acid Tablets, 300 mg Lot O

This USP Dissolution Calibrator is provided for the *Apparatus Suitability Test* in the General Chapters (711) and (724). **Do not expose to excessive humidity.**

Procedure—[See *Dissolution* (711) and *Drug Release* (724) in the current USP.] Determine the quantity of salicylic acid, $C_7H_6O_3$, dissolved at thirty minutes, for each spindle, expressed as percent of the labeled amount. Use 900 mL of deaerated 0.05 M phosphate buffer pH 7.40 ± 0.05 (pH tested at room temperature) as the *Dissolution Medium*. The test is conducted at 37° and the apparatus are operated at each of the speeds indicated in the Table below. Measure the amount of salicylic acid in solution in filtered portions of the *Dissolution Medium*, suitably diluted with fresh *Dissolution Medium*, if necessary, at the wavelength of maximum absorbance at about 296 nm in comparison with a solution of known concentration of USP Salicylic Acid Reference Standard.

Test Interpretation—The apparatus is suitable if each of the individual calculated values for each apparatus at all indicated speeds are within the specified ranges, as shown in the Table.

Notes: An amount of alcohol not to exceed 1% of the total volume of the standard solution may be used to bring the salicylic acid standard into solution prior to dilution with *Dissolution Medium*. Filtering method must be checked for adsorptive loss of drug. In the case of membrane filters, do not use the first 2 mL of solution unless separate interference and recovery experiments have been carried out. Bias introduced by automated methods is to be avoided. These tablets are pure salicylic acid with no binders or fillers. Because of the physical properties of such tablets some sticking of the tablets may occur during storage. Gentle pressure or tapping of the bottle may be used to separate tablets. Cracked, "capped", or severely chipped tablets should not be used. However, tablets with minor surface flaws are generally acceptable for use. Powder on the surface of tablets should be removed prior to use of the tablets. **If equipment is dedicated for use with only one apparatus (basket or paddle), then the calibration is not required for both apparatus.**

See other side for helpful suggestions.

These values apply only to Lot O

Apparatus	% dissolved at 30 minutes	
	50 rpm	100 rpm
1	--	23-29
2	--	17-26

Founded in 1820, the United States Pharmacopeial Convention comprises representatives from colleges and national and state organizations of medicine and pharmacy. It revises and publishes *The United States Pharmacopeia* and *The National Formulary*, the legally recognized compendia of standards for drugs.

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Salicylic Acid Tablets: Calibration Notes

Dissolution equipment that has been routinely used for a number of years (three-five) should be serviced if out-of-range values are obtained. Any dissolution equipment that is used routinely should be calibrated at regular intervals. Relocation of apparatus always requires recalibration. Some USP Dissolution tests require 2-L vessels or speeds other than 50 and 100 rpm. The equipment is suitable for these other conditions if it passes the calibration tests.

Sources of Error in Calibration Testing

Deaeration of medium. Improper deaeration is a common problem. This formulation has been demonstrated to be exquisitely sensitive to dissolved gases in the medium. The deaeration method used in the testing to establish the certificate ranges is as follows: Heat a suitable volume of water to 41° and filter with the aid of vacuum through a 0.45- μ m-porosity membrane into a suitable filtering flask, equipped with a stirring device. Seal the flask and continue to apply a vacuum while stirring for an additional five minutes. Do not allow the temperature of the *Dissolution Medium* to fall below 37° prior to the initiation of the test. Gently transfer the Medium directly to the vessel. Do not introduce air into the Medium. Rotating the Apparatus 2 shafts to speed equilibration to 37° is discouraged. Use medium promptly after it is equilibrated.

Vessels. Vessels must be clean. Use of an unacceptable vessel is a systematic error.

Vibration and mechanical problems. When not properly examined and maintained, factors such as dissolution head coplanarity, shaft perpendicularity, tension on the drive chain or belt, centering, and operating condition of the gear plates can adversely affect dissolution. Digital rpm readings may not necessarily represent individual spindle speeds. Visual inspection may be needed to observe surging of the separate spindles. To minimize vibration effects, the dissolution equipment should be on a stable benchtop or table. Other mechanical equipment using fans, pumps, or other vibration sources should be removed from the area or isolated in some other way. Turbulence in the waterbath caused by circulation patterns can affect results in one or more vessels.

Automation. Always validate the automated method, including the analytical method and sampling method, by performing a parallel manual analysis, withdrawing test samples at the same times, and comparing to the automated results. Filter probes may become clogged, absorb the active ingredient, or generate additional turbulence through the air-purging step. Be alert to the possibility of carryover among samplings. Automated systems may not account for dilution and the absorbance reading may be over 1.0 absorbance units. Linearity above 1.0 absorbance should be established with a standard curve.

Tablets. The Calibrator Tablets should be stored in the original containers in a dry place. Avoid excess humidity. When testing, take the tablets from the bottle and begin the dissolution test immediately.

Reference Standard. Use the current lot of USP Reference Standard and follow any drying instructions on the label. Prepare the standard solution on the day of use.

Filtering. Do not centrifuge sample. The sample aliquot should be filtered immediately after the sample is drawn. The filters should be tested for interference from leachables or by adsorption of the drug. A separate clean syringe and filter should be used for sampling each vessel.

Paddles and baskets. The shafts of both apparatuses should be straight. A simple test of this is to roll the shaft on the bench top with the paddle blade or prongs for the basket hanging over the edge. The shaft should roll evenly like an arrow shaft. Baskets should be straight and not frayed. Routine use in hydrochloric acid Medium causes deterioration of the stainless steel baskets. Baskets should attach firmly to the shaft prongs. Evaporation lids should be used. Inspect them for fit or warping.