U.S. Pharmacopeia
The Standard of Quality℠

USP Certificate

USP Salicylic Acid Tablets RS
Lot Q0D200
(300 mg nominal salicylic acid content per tablet)

USP DISSOLUTION CALIBRATOR,
NON-DISINTEGRATING TYPE

This USP Dissolution Calibrator is provided for use in the **Apparatus Suitability Test** for USP Apparatus 1 and 2 in the USP General Test Chapters on DISSOLUTION <711> and DRUG RELEASE <724>. Do not expose the tablets to excessive humidity.

Dissolution Medium- We recommend preparing the medium as follows:

Transfer a 41-g portion of monobasic potassium phosphate into a 6-L volumetric flask, and dissolve in 5.8 L of water. Add portions of sodium hydroxide solution 50% (w/w) to obtain a pH of 7.4 ± 0.05. Dilute this solution with water to volume. Readjust the pH if necessary with sodium hydroxide or phosphoric acid. Another method to prepare a pH 7.4 Phosphate Buffer Solution is described in the USP Chapter for Solutions. Deaerate the *Dissolution Medium* with a technique validated for 37º. One method of deaeration is described in the USP General Test Chapter on DISSOLUTION <711>.

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, in each vessel, expressed as percent of the labeled amount. Use 900 mL of 0.05*M* Phosphate Buffer pH=7.4 ± 0.05 (pH tested at room temperature) as the *Dissolution Medium* and conduct the test at 37°. Operate each apparatus at 100-rpm speed. Measure the amount of salicylic acid dissolved from filtered portions of the sample aliquots withdrawn and suitably diluted with *Dissolution Medium* if necessary at 296 nm (the approximate wavelength of maximum absorbance) in comparison with a solution of known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 is within the specified ranges 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*. The filtering method must not cause adsorptive loss of drug. Bias introduced by automated methods is to be avoided. 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 only required for that apparatus.
See other side for helpful suggestions.

These values apply only to Lot Q0D200

Apparatus	**Percentage of the labeled amount of salicylic acid dissolved at 30 minutes at 100 rpm**
1	**23-30**
2	**17-25**

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.

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 sensitive to dissolved gases in the medium**. One method of deaeration is as follows: Heat the medium, while stirring gently, to about 41º, and filter under vacuum through a 0.45-µm-porosity membrane into a suitable filtering flask, equipped with a stirring device. Seal the flask and continue to apply 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. 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 bench top 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 water bath 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 blister pack 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.

LABEL TEXT

USP certifies that the USP Reference Standards Committee, in accordance with their rules and procedures, determined that this USP Reference Standard lot is suitable to assess compliance with the monograph standards for which it is specified. The critical characteristics of this lot are usually determined independently in a large number of laboratories, including USP, government, academic, and industrial collaborators.

QA Director

Expiration

Current lots are identified in the Official USP Reference Standards catalog. In some cases, the previous lot may still be considered official. If so, it is identified in the column marked "Previous Lot/Valid Use Date." Ordinarily, the previous lot is carried in official status for about one year after the current lot enters distribution.

It is the responsibility of each user to determine that this lot is current when used. To ensure up-to-date information, USP publishes the Official USP Reference Standards Catalog, which contains official lot designations. This information is also available on the USP web site, at www.usp.org, as well as in the bimonthly subscription publication, *Pharmacopeial Forum*.

Instructions for Use

Follow the instructions in the appropriate USP or NF Monographs and General Requirements for Tests and Assays of the current *USP–NF*. In the event that instructions on the label of this lot differ from those found in the current *USP–NF*, those on the label supersede any instructions listed in Chapter <11>.

Non-Monograph Use

The suitability of this Reference Standard for use in non-compendial applications is solely the responsibility of the user.
