



U.S. Pharmacopeia
The Standard of QualitySM

Effective date: December 6, 2004

NOTE: The specifications in this sheet supercede the previous specifications issued for this lot.

**USP DISSOLUTION CALIBRATOR,
DISINTEGRATING TYPE**

**USP Prednisone Tablets RS
Lot O0C056
(10 mg nominal prednisone content per tablet)**

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- Heat a suitable amount of water, while stirring gently, to about 41°. Filter under 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 vacuum while stirring for an additional five minutes. The temperature of the *Dissolution medium* does not fall below 37° prior to the initiation of the test.

Procedure- [See DISSOLUTION <711> and DRUG RELEASE <724> in the current USP.] Determine the quantity of prednisone, C₂₁H₂₆O₅, dissolved at thirty minutes, in each vessel, expressed as percent of the labeled amount. Use 500 mL of deaerated water as the *Dissolution medium* and conduct the test at 37°. Operate each apparatus at 50- rpm speed. Measure the amount of prednisone dissolved from filtered portions of the sample aliquots withdrawn at thirty minutes, at the wavelength of maximum absorbance at about 242 nm in comparison with a solution of known concentration of USP Prednisone Reference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 5% of the total volume of the standard solution may be used to bring the prednisone 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. If equipment is dedicated for use with only one apparatus (basket or paddle), then the calibration is only required for that apparatus.

These values apply only to Lot O0C056

Apparatus	Percentage of the labeled amount of prednisone dissolved at 30 minutes at 50-rpm
1	51-81
2	26-47

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.

12601 Twinbrook Parkway
Rockville, MD 20852

301-881-0666

www.usp.org



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Prednisone 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 the Dissolution Medium to about 41°. Vacuum filter through a 0.45- μ m-porosity membrane into a 4-liter filter flask, stirring with a magnetic stirrer. Continue to draw a vacuum and stir for an additional 5 minutes. 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.