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Thalidomide Capsules

» Thalidomide Capsules contain not less than 90.0 percent and not more than 110.0 percent of the labeled amount of thalidomide ($C_{13}H_{10}N_2O_4$).

Packaging and storage—Preserve in tight containers, protected from light, at controlled room temperature. Do not repackage.

USP Reference standards ⟨11⟩—

USP Thalidomide RS

Identification—

A: *Thin-Layer Chromatographic Identification Test ⟨201⟩*—

Test solution—Prepare a solution of it in acetonitrile containing about 3000 µg of thalidomide per mL.

Application volume: 5 µL.

Developing solvent system: a mixture of normal butyl acetate, glacial acetic acid, and butyl alcohol (50:25:5).

B: The relative retention time of the major peak in the chromatogram of the *Assay preparation* corresponds to that in the chromatogram of the *Standard preparation,* as obtained in the *Assay.*

Microbial enumeration tests ⟨61⟩ and Tests for specified microorganisms ⟨62⟩—The total aerobic microbial count using the *Plate Method* is not more than 1000 cfu per g, and the total combined molds and yeasts count is not more than 100 cfu per g. It meets the requirements of the test for absence of *Escherichia coli.*

Dissolution ⟨711⟩—

Medium—Add 1.0 mL of polyoxyethylene (23) lauryl ether solution, prepared by dissolving 50 g in 100 mL of water, to 0.225 M hydrochloric acid; 4000 mL.

Apparatus 2: 75 rpm.

Time: 60 minutes.

Determine the amount of $C_{13}H_{10}N_2O_4$ dissolved by employing the following method.

Mobile phase—Prepare as directed in the *Assay* under *Thalidomide*.

Internal standard solution—Prepare a solution of phenacetin in acetonitrile containing about 375 µg per mL. Pipet 20.0 mL of this solution into a 100-mL volumetric flask, add 10.0 mL of phosphoric acid solution (1 in 100), dilute with water to volume, and mix.

Standard solution—Dissolve an accurately weighed quantity of USP Thalidomide RS in acetonitrile to obtain a solution having a known concentration of about 0.25 mg per mL. Pipet 10.0 mL of this solution into a 100-mL volumetric flask, add 10.0 mL of phosphoric acid solution (1 in 100), dilute with water to volume, and mix. Add 5.0 mL of *Internal standard solution* to 20.0 mL of this solution, and mix. This solution contains about 0.02 mg of USP Thalidomide RS per mL.

Test solution—Add 5.0 mL of *Internal standard solution* to each 20.0 mL of filtered solution under test, and mix.

Chromatographic system—Prepare as directed in the *Assay* under *Thalidomide*.

Procedure—Separately inject equal volumes (about 20 µL) of the *Standard solution* and the *Test solution* into the chromatograph, record the chromatograms, and measure the areas for the major peaks. Calculate the quantity, in mg, of $C_{13}H_{10}N_2O_4$ dissolved by the formula:

$$2500C(R_U/R_S)$$

in which C is the concentration, in mg per mL, of USP Thalidomide RS in the *Standard solution;* and R_U and R_S are the peak area ratios of thalidomide to the internal standard obtained from the *Test solution* and the *Standard solution*, respectively.

Tolerances—Not less than 70% *(Q)* of the labeled amount of $C_{13}H_{10}N_2O_4$ is dissolved in 60 minutes.

Uniformity of dosage units ⟨905⟩: meet the requirements.

Assay—

Mobile phase, *Internal standard preparation*, *Standard preparation*, and *Chromatographic system*—Prepare as directed in the *Assay* under *Thalidomide.*

Assay preparation—Remove, as completely as possible, the contents of not fewer than 20 Capsules, and weigh accurately. Mix the combined contents, and transfer an accurately weighed portion of the powder, equivalent to about 50 mg of thalidomide, to a 100-mL volumetric flask, add 80 mL of acetonitrile to dissolve, and sonicate for about 20 minutes. Dilute with acetonitrile to volume, and mix. Transfer 20.0 mL of this solution and 5.0 mL of *Internal standard preparation* to a 100-mL volumetric flask, add 10.0 mL of phosphoric acid solution (1 in 100), dilute with water to volume, and mix.

Change to read:

Procedure—Separately inject equal volumes (about 20 µL) of the *Standard preparation* and the *Assay preparation* into the chromatograph, record the chromatograms, and measure the areas for the major peaks. Calculate the quantity, in mg, of thalidomide ($C_{13}H_{10}N_2O_4$) in the portion of Capsules taken by the formula:

$$▲500▲ \text{ (ERR 1-Mar-2024) } C(R_U/R_S)$$

in which C is the concentration, in mg per mL, of USP Thalidomide RS in the *Standard preparation;* and R_U and R_S are the peak area ratios of thalidomide to the internal standard obtained from the *Assay preparation* and the *Standard preparation,* respectively.

Auxiliary Information - Please check for your question in the FAQs before contacting USP.

Topic/Question	Contact	Expert Committee
THALIDOMIDE CAPSULES	Documentary Standards Support	SM32020 Small Molecules 3
REFERENCE STANDARD SUPPORT	RS Technical Services RSTECH@usp.org	SM32020 Small Molecules 3

Chromatographic Database Information: Chromatographic Database

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