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## <451> NITRITE TITRATION

The following general method is provided for the determination of most of the Pharmacopeial sulfonamide drugs and their dosage forms, as well as of other Pharmacopeial drugs for which nitrite titration is particularly suitable.

### PROCEDURE

Accurately weigh about 500 mg in the case of a sulfonamide, or otherwise the quantity specified in the individual monograph, and transfer to a suitable open vessel. Add 20 mL of hydrochloric acid and 50 mL of water, stir until dissolved, cool to about 15°, and slowly titrate with 0.1 M sodium nitrite VS that previously has been standardized against [USP Sulfanilamide RS](#).

Determine the endpoint electrometrically, using suitable electrodes (platinum-calomel or platinum-platinum). Place the buret tip below the surface of the solution to eliminate air oxidation of the sodium nitrite, and stir the solution gently, using a magnetic stirrer, without pulling a vortex of air under the surface, maintaining the temperature at about 15°. The titration may be carried out manually, or by means of an automatic titrator. In performing it manually, add the titrant until the titration is within 1 mL of the endpoint, and then add it in 0.1-mL portions, allowing not less than 1 minute between additions. (The instrument needle deflects and then returns to approximately its original position until the endpoint is reached.)

The weight, in mg, of the substance to which each mL of 0.1 M sodium nitrite VS is equivalent is as stated in the individual monograph.

For the assay of Tablets of the sulfonamides or other drugs, reduce not less than 20 tablets to a fine powder, weigh accurately a portion of the powder, equivalent to about 500 mg if a sulfonamide, or the quantity of drug specified in the individual monograph, and proceed as directed in the foregoing, beginning with "transfer to a suitable open vessel."

For the assay of Injections and other liquid forms where the nitrite titration is specified, pipet a portion, equivalent to about 500 mg if a sulfonamide, or the quantity of drug specified in the individual monograph, into a suitable open vessel, and proceed as directed in the foregoing, beginning with "Add 20 mL of hydrochloric acid."

### ADDITIONAL REQUIREMENTS

- [USP REFERENCE STANDARDS \(11\)](#)  
[USP Sulfanilamide RS](#)

**Auxiliary Information** - Please [check for your question in the FAQs](#) before contacting USP.

| Topic/Question          | Contact   | Expert Committee                                   |
|-------------------------|---|--|
| <451> NITRITE TITRATION | <a href="#">Yang Liu</a><br>Manager, Product Quality and Analytical Methods | GCCA2020 General Chapters - Chemical Analysis 2020 |

### Most Recently Appeared In:

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