

**Assay preparation**—Using water as the diluent, quantitatively dilute an accurately measured volume of *Injection* to obtain a solution containing about 40 µg of selenium per mL.

**Procedure**—Concomitantly determine the absorbances of the *Standard preparations* and the *Assay preparation* at the selenium emission line of 196 nm, with a suitable atomic absorption spectrophotometer (see *Spectrophotometry and Light-scattering* <851>) equipped with a selenium electrodeless discharge lamp and an air-acetylene flame, using water as the blank. Plot the absorbances of the *Standard preparations* versus concentration, in µg per mL, of selenium, and draw the straight line best fitting the three plotted points. From the graph so obtained, determine the concentration *C*, in µg per mL, of selenium in the *Assay preparation*. Calculate the quantity, in mg, of selenium in each mL of the *Injection* taken by the formula:

$$LC / D$$

in which *L* is the labeled quantity, in mg per mL, of selenium in the *Injection* taken, and *D* is the concentration, in µg of selenium per mL, of the *Assay preparation* on the basis of the labeled quantity in the *Injection* and the extent of dilution.

## Selenium Sulfide

SeS<sub>2</sub> 143.09

Selenium sulfide (SeS<sub>2</sub>).

Selenium sulfide (SeS<sub>2</sub>) [7488-56-4].

» Selenium Sulfide contains not less than 52.0 percent and not more than 55.5 percent of selenium (Se).

**Packaging and storage**—Preserve in well-closed containers.

### Identification

**A:** Filter 20 mL of the solution of Selenium Sulfide prepared as directed in the *Assay*, and to 10 mL of the filtrate add 5 mL of water and 5 g of urea. Heat to boiling, cool, and add 2 mL of potassium iodide solution (1 in 10); a yellowish orange to orange color is produced, and it darkens rapidly (*presence of selenium*).

**B:** Allow the solution obtained in *Identification test A* to stand for 10 minutes, filter, and to the filtrate add 10 mL of barium chloride TS; the solution becomes turbid (*presence of sulfur*).

**Residue on ignition** <281>: not more than 0.2%.

### Soluble selenium compounds

**Test solution**—Mix 10.0 g of Selenium Sulfide with 100.0 mL of water in a 250-mL flask, allow to stand for 1 hour, with frequent agitation, and filter. To 10.0 mL of the filtrate add 2 mL of 2.5 M formic acid, dilute with water to 50 mL, mix, and adjust, if necessary, to a pH of 2.5 ± 0.5. Add 2 mL of freshly prepared 3,3'-diaminobenzidine hydrochloride solution (1 in 200), mix, allow to stand for 45 minutes, and adjust with 6 N ammonium hydroxide to a pH of 6.5 ± 0.5. Transfer to a separator, add 10.0 mL of toluene, shake vigorously for 1 minute, allow the layers to separate, and discard the aqueous phase.

**Standard solution**—Using 10.0 mL of a solution of selenious acid containing 0.5 µg of selenium per mL, prepare a solution as directed under *Test solution*, beginning with “add 2 mL of 2.5 M formic acid.”

**Procedure**—Concomitantly determine the absorbances of the toluene layers of the *Test solution* and the *Standard solution* in 1-cm cells at 420 nm, with a suitable spectrophotometer, using a blank consisting of the same quantities of the same reagents treated in the same manner as the *Test solution*; the absorbance of the *Test solution* is not greater than that of the *Standard solution* (5 ppm).

**Assay**—Place about 100 mg of Selenium Sulfide, accurately weighed, in a suitable container, add 25 mL of fuming nitric acid, and digest over gentle heat until no further solution occurs. Cool, transfer the solution to a 250-mL volumetric flask containing 100 mL of water, cool again, dilute with water to volume, and mix. Pipet 50 mL of the solution into a suitable flask, add 25 mL of water and 10 g of urea, and heat to boiling. Cool, add 3 mL of starch TS, then add 10 mL of potassium iodide solution (1 in 10), and immediately titrate with 0.05 N sodium thiosulfate VS. Perform a blank determination, and make any necessary correction. Each mL of 0.05 N sodium thiosulfate is equivalent to 987.0 µg of Se.

## Selenium Sulfide Topical Suspension

» Selenium Sulfide Topical Suspension is an aqueous, stabilized suspension of Selenium Sulfide. It contains not less than 90.0 percent and not more than 110.0 percent of the labeled amount of SeS<sub>2</sub>. It contains suitable buffering and dispersing agents.

**NOTE**—Where labeled for use as a shampoo, it contains a detergent. Where labeled for other uses, it may contain a detergent.

**Packaging and storage**—Preserve in tight containers.

**Identification**—Digest about 2 g with 5 mL of nitric acid over gentle heat for 1 hour, dilute with water to about 50 mL, and filter; the solution responds to *Identification test A* under *Selenium Sulfide*, when tested as directed, beginning with “to 10 mL of the filtrate add 5 mL of water.”

**pH** <791>: between 2.0 and 6.0.

**Assay**—Place a portion of well-mixed Topical Suspension, equivalent to about 100 mg of selenium sulfide and accurately weighed, in a suitable flask. Cautiously digest with 25 mL of fuming nitric acid over gentle heat for 2 hours, and proceed as directed in the *Assay* under *Selenium Sulfide*, beginning with “Cool, transfer the solution to a 250-mL volumetric flask.” Each mL of 0.05 N sodium thiosulfate is equivalent to 1.789 mg of SeS<sub>2</sub>. Where the Topical Suspension is labeled in terms of percentage (w/v) or of the amount of SeS<sub>2</sub> in a given volume of Topical Suspension, determine the density of the Topical Suspension as follows: Using a tared, 100-mL volumetric flask, weigh 100 mL of Topical Suspension that previously has been shaken to ensure homogeneity, allowed to stand until the entrapped air rises, and finally inverted carefully just prior to transfer to the volumetric flask. From the observed weight of 100 mL of the Topical Suspension, calculate the quantity of SeS<sub>2</sub> in each 100 mL.

## Senna Leaf

### DEFINITION

Senna Leaf consists of the dried leaflet of *Senna alexandrina* Mill. also known as *Cassia acutifolia* Delile (Alexandrian senna) or *C. angustifolia* Vahl (Tinnevelly senna) (Fam. Fabaceae). Senna Leaf contains NLT 2.5% of anthraquinone glucosides, calculated as sennosides, on the dried basis.

### IDENTIFICATION

#### • A.

**Potassium hydroxide solution:** 100 mg/mL of potassium hydroxide in alcohol

**Sample:** 500 mg of finely powdered Senna Leaf

**Analysis:** Add 10 mL of *Potassium hydroxide solution* to the *Sample*. Boil for 2 min, dilute with 10 mL of water, and filter. Acidify the filtrate with hydrochloric acid. Shake it with