Solution B: 10  $\mu$ L of the *Standard Lead Solution*, 10  $\mu$ L of the *Sample Preparation*, 10  $\mu$ L of the *Blank Solution*, and 5  $\mu$ L of the *Modifier Working Solution*;

Solution C: 20  $\mu$ L of the *Standard Lead Solution*, 10  $\mu$ L of the *Sample Preparation*, and 5  $\mu$ L of the *Modifier Working Solution*;

Solution D: 10  $\mu$ L of the Sample Preparation, 20  $\mu$ L of the Blank Solution, and 5  $\mu$ L of the Modifier Working Solution.

Calculate the blank-corrected absorbances of Solutions B, C, and D by subtracting from each the absorbance measured for Solution A. Plot the blank-corrected absorbances of Solutions B, C, and D (y-axis) versus the quantity of lead, in nanograms, added to each solution (x-axis). These are equal to 0.1, 0.2, and 0 ng, respectively. Draw the best straight line through the points. Extrapolate the line to the x-axis intercept to obtain the quantity *C*, in nanograms, of lead in 10  $\mu$ L of the *Sample Solution*. Calculate the concentration, in milligrams per kilogram, of lead in the sample taken by the formula

#### 10*C/W*,

in which *W* is the weight, in grams, of the sample taken. **Optical (Specific) Rotation** Determine as directed under *Optical (Specific) Rotation*, Appendix IIB, using a solution containing 2 g of sample in 100 mL of oxygen-free water.

**Oxalic Acid** Dissolve 1 g of sample in 10 mL of water and 2 mL of hydrochloric acid, transfer to a separator, and extract with two 35-mL portions of ether. Evaporate the combined ether extracts in a rotary evaporator or on a steam bath. Dissolve any residue in 10 mL of water, add 1 mL of glacial acetic acid and 1 mL of a 1:20 solution of calcium acetate. No turbidity develops in 5 min.

**Sulfate** Determine as directed in the *Sulfate Limit Test* under *Chloride and Sulfate Limit Tests*, Appendix IIIB. Any turbidity produced by a 20-mL portion of the solution prepared for the *Chloride Test* (above) does not exceed that shown in a control containing 200  $\mu$ g of sulfate (SO<sub>4</sub>).

**Water** Determine as directed under *Water Determination*, Appendix IIB, at 50°, using 100 mg of sample dissolved in a freshly prepared mixture of 20 mL of methanol and 20 mL of formamide.

Packaging and Storage Store in tight containers.

## **Ferrous Sulfate**

 $FeSO_4 \cdot 7H_2O$ 

Formula wt 278.02 CAS: [7782-63-0]

#### DESCRIPTION

Ferrous Sulfate occurs as pale, blue-green crystals or granules that are efflorescent in dry air. In moist air, it oxidizes readily to form a brown-yellow, basic ferric sulfate. A 1:10 aqueous solution has a pH of about 3.7. One gram dissolves in 1.5

Function Nutrient.

#### REQUIREMENTS

**Identification** A sample gives positive tests for *Ferrous Salts* (Iron) and for *Sulfate*, Appendix IIIA.

**Assay** Not less than 99.5% and not more than 104.5% of  $FeSO_4$ ·7H<sub>2</sub>O.

**Lead** Not more than 2 mg/kg.

**Mercury** Not more than 1 mg/kg.

#### TESTS

Assay Dissolve about 1 g of sample, accurately weighed, in a mixture of 25 mL of 2 N sulfuric acid and 25 mL of recently boiled and cooled water, and immediately titrate with 0.1 N ceric sulfate, using orthophenanthroline TS as the indicator. Perform a blank determination (see *General Provisions*), and make any necessary correction. Each milliliter of 0.1 N ceric sulfate is equivalent to 27.80 mg of FeSO<sub>4</sub>·7H<sub>2</sub>O.

**Lead** Determine as directed in the monograph for *Ferrous Gluconate*.

**Mercury** Determine as directed in *Method II* under *Mercury Limit Test*, Appendix IIIB.

Packaging and Storage Store in tight containers.

## Ferrous Sulfate, Dried

FeSO <sub>4</sub> · <i>x</i> H <sub>2</sub> O	Formula wt, anhydrous 151.91
	CAS: [7720-78-7]

#### DESCRIPTION

Ferrous Sulfate, Dried, occurs as a gray-white to buff colored powder consisting primarily of  $FeSO_4$ ·H<sub>2</sub>O, with varying amounts of  $FeSO_4$ ·4H<sub>2</sub>O. It dissolves slowly in water, but is insoluble in alcohol.

Function Nutrient.

#### REQUIREMENTS

**Identification** A sample gives positive tests for *Ferrous Salts* (Iron) and for *Sulfate*, Appendix IIIA.

Assay Not less than 86.0% and not more than 89.0% of FeSO<sub>4</sub>.

**Insoluble Substances** Not more than 0.05%.

**Lead** Not more than 2 mg/kg.

**Mercury** Not more than 1 mg/kg.

#### TESTS

**Assay** Determine as directed under *Assay* in the monograph for *Ferrous Sulfate*. Each milliliter of 0.1 N ceric sulfate is equivalent to 15.19 mg of FeSO<sub>4</sub>.

**Insoluble Residue** Dissolve 2 g of sample in 20 mL of freshly boiled 1:100 sulfuric acid, heat to boiling, and then digest in a covered beaker on a steam bath for 1 h. Filter through a tared filtering crucible, wash thoroughly, and dry at  $105^{\circ}$ . The weight of the insoluble residue does not exceed 1 mg.

**Lead** Determine as directed in the monograph for *Ferrous Gluconate*.

**Mercury** Determine as directed in *Method II* under *Mercury Limit Test*, Appendix IIIB.

Packaging and Storage Store in tight containers.

Fir Needle Oil, Canadian Type

Balsam Fir Oil

### DESCRIPTION

Fir Needle Oil, Canadian Type, occurs as a colorless to faintly yellow liquid with a pleasant, balsamic odor. It is the volatile oil obtained by steam distillation from needles and twigs of *Abies balsamea* L., Mill (Fam. Pinaceae). It is soluble in most fixed oils and in mineral oil. It is slightly soluble in propylene glycol, but it is insoluble in glycerin.

Function Flavoring agent.

#### REQUIREMENTS

**Identification** The infrared absorption spectrum of the sample exhibits relative maxima at the same wavelengths as those of a typical spectrum as shown in the section on *Infrared Spectra*, using the same test conditions as specified therein. **Assay** Not less than 8.0% and not more than 16.0% of esters, calculated as bornyl acetate  $(C_{12}H_{20}O_2)$ .

Angular Rotation Between -19° and -24°. Refractive Index Between 1.473 and 1.476 at 20°. Solubility in Alcohol Passes test. Specific Gravity Between 0.872 and 0.878.

## TESTS

**Assay** Measure about 5 g of sample, accurately weighed, and proceed as directed in *Ester Determination* under *Esters*, Appendix VI, using 98.15 as the equivalence factor (e) in the calculation.

**Angular Rotation** Determine as directed under *Optical* (*Specific*) *Rotation*, Appendix IIB, using a 100-mm tube.

**Refractive Index** Determine as directed under *Refractive Index*, Appendix IIB, using an Abbé or other refractometer of equal or greater accuracy.

**Solubility in Alcohol** Determine as directed under *Solubility in Alcohol*, Appendix VI. One milliliter of sample dissolves in 4 mL of 90% alcohol, occasionally with haziness.

**Specific Gravity** Determine by any reliable method (see *General Provisions*).

**Packaging and Storage** Store in a cool place protected from light in full, tight containers that are made from steel or aluminum and that are suitably lined.

# Fir Needle Oil, Siberian Type

Pine Needle Oil

View IR

### DESCRIPTION

**View IR** 

Fir Needle Oil, Siberian Type, occurs as an almost colorless or faintly yellow liquid with a piney, balsamic odor. It is the volatile oil obtained by steam distillation from needles and twigs of *Abies sibirica* Lebed. (Fam. Pinaceae). It is soluble in most fixed oils and in mineral oil. It is insoluble in glycerin and in propylene glycol.

**Function** Flavoring agent.

## REQUIREMENTS

**Identification** The infrared absorption spectrum of the sample exhibits relative maxima at the same wavelengths as those of a typical spectrum as shown in the section on *Infrared Spectra*, using the same test conditions as specified therein. **Assay** Not less than 32.0% and not more than 44.0% of esters, calculated as bornyl acetate ( $C_{12}H_{20}O_2$ ). **Angular Rotation** Between  $-33^{\circ}$  and  $-45^{\circ}$ . **Refractive Index** Between 1.468 and 1.473 at 20°. **Solubility in Alcohol** Passes test.

**Specific Gravity** Between 0.898 and 0.912.

## TESTS

**Assay** Measure about 2 g of sample, accurately weighed, and proceed as directed in *Ester Determination* under *Esters*, Appendix VI, using 98.15 as the equivalence factor (e) in the calculation.

**Angular Rotation** Determine as directed under *Optical* (*Specific*) *Rotation*, Appendix IIB, using a 100-mm tube.

**Refractive Index** Determine as directed under *Refractive Index*, Appendix IIB, using an Abbé or other refractometer of equal or greater accuracy.

**Solubility in Alcohol** Determine as directed under *Solubility in Alcohol*, Appendix VI. One milliliter of sample dissolves in 1 mL of 90% alcohol. Occasionally the solution may become hazy on further dilution.