sium iodide TS and 20 mL of water. Assemble the apparatus immediately, and stir while slowly adding through the side arm of the flask, over a period of about 20 minutes, 40 mL of *Sodium borohydride solution*. Examine the stain produced on the *Mercuric bromide paper*. Perform the same procedure using the *Standard solution*. The stain produced on the *Mercuric bromide paper* from the *Test solution* is not more intense than that from the *Standard solution* (3 µg per g).

Limit of lead—

Lead nitrate stock solution—Transfer 159.8 mg of lead nitrate to a 100-mL volumetric flask, dissolve in and dilute with 0.5 M nitric acid to volume, and mix. Prepare and store this solution in glass containers free from soluble lead salts.

Standard solution—On the day of use, transfer 5.0 mL of the *Lead nitrate stock solution* to a 100-mL volumetric flask, add 10 mL of 1 N hydrochloric acid, dilute with water to volume, and mix. Transfer 1.0 mL of this solution to a second 100-mL volumetric flask, add 10 mL of 1 N hydrochloric acid, dilute with water to volume, and mix. This solution contains 0.5 µg of lead per mL.

Test solution—Transfer 2.5 g of Ferric Oxide to a 100-mL glass-stoppered conical flask, add 35 mL of 0.1 N hydrochloric acid, and stir for 1 hour. Filter, collecting the filtrate in a 50-mL volumetric flask, dilute with 0.1 N hydrochloric acid to volume, and mix.

Procedure—Concomitantly determine the absorbance of the *Standard solution* and the *Test solution* at the lead emission line of 217.0 nm with an atomic absorption spectrophotometer (see *Spectrophotometry and Light-Scattering* ⟨851⟩) equipped with a lead hollow-cathode lamp, a flow spoiler, and an air–acetylene oxidizing flame. The absorbance of the *Test solution* does not exceed that of the *Standard solution* (0.001%).

Assay—Digest about 1.5 g of Ferric Oxide, accurately weighed, in 25 mL of hydrochloric acid on a water bath until dissolved. Add 10 mL of hydrogen peroxide TS, and evaporate on a water bath almost to dryness in order to volatilize all hydrogen peroxide. Dissolve the residue by warming with 5 mL of hydrochloric acid; add 25 mL of water; filter into a 250-mL volumetric flask, washing the filter with water; and add water to volume. Transfer a 50-mL aliquot to a glass-stoppered flask, add 3 g of potassium iodide and 5 mL of hydrochloric acid, and insert the stopper into the flask. Allow the mixture to stand for 15 minutes, add 50 mL of water, and titrate the liberated iodine with 0.1 N sodium thiosulfate VS, using starch TS as the indicator. Perform a blank test with the same quantities of reagents and in the same manner, and make any necessary corrections. Each mL of 0.1 N sodium thiosulfate is equivalent to 7.985 mg of Fe_2O_3. Ignite about 2 g of Ferric Oxide at 800 ± 25° to constant weight as directed under *Loss on Ignition* ⟨733⟩, to enable calculation of the percentage of Fe_2O_3 on the ignited basis. [NOTE—Ignited Ferric Oxide is hygroscopic.]

Fructose—*see Fructose General Monographs*

Fumaric Acid

$C_4H_4O_4$ 116.07
2-Butenedioic acid, *E*-;
Fumaric acid [110-17-8].

DEFINITION

Fumaric Acid contains NLT 99.5% and NMT 100.5% of $C_4H_4O_4$, calculated on the anhydrous basis.

IDENTIFICATION

- **INFRARED ABSORPTION** ⟨**197A**⟩

ASSAY

- **PROCEDURE**

Sample solution: Dissolve 1 g of Fumaric Acid in 50 mL of methanol, and warm gently on a steam bath to effect solution. Cool, and add phenolphthalein TS.

Analysis: Titrate the *Sample solution* with 0.5 N sodium hydroxide VS to the first appearance of a pink color that persists for not less than 30 s. Perform a blank titration (see *Titrimetry* ⟨541⟩). Each mL of 0.5 N sodium hydroxide is equivalent to 29.02 mg of $C_4H_4O_4$.

Acceptance criteria: 99.5%–100.5% on the anhydrous basis

IMPURITIES

Inorganic Impurities

- **RESIDUE ON IGNITION** ⟨**281**⟩**:** NMT 0.1%
- **HEAVY METALS,** *Method II* ⟨**231**⟩**:** NMT 10 ppm

Organic Impurities

- **PROCEDURE: LIMIT OF MALEIC ACID**

Mobile phase: 0.005 N sulfuric acid

Standard solution: 1 µg/mL of USP Maleic Acid RS in *Mobile phase*

Sample solution: 1 mg/mL of Fumaric Acid in *Mobile phase*

System suitability solution: 10 µg/mL of USP Fumaric Acid RS and 5 µg/mL of USP Maleic Acid RS in *Mobile phase*

Chromatographic system

(See *Chromatography* ⟨621⟩*, System Suitability*.)

Mode: LC

Detector: UV 210 nm

Column: 4.6-mm × 22-cm; packing L17

Flow rate: 0.3 mL/min

Injection size: 5 µL

System suitability

Sample: *System suitability solution*

[NOTE—The relative retention times for maleic acid and fumaric acid are about 0.5 and 1.0, respectively.]

Suitability requirements

Resolution: NLT 2.5 of the maleic acid and fumaric acid peaks

Relative standard deviation: NMT 2.0% of the maleic acid peak

Analysis

Samples: *Standard solution* and *Sample solution*

Calculate the percentage of maleic acid in the total weight of Fumaric Acid taken:

$$\text{Result} = (r_U/r_S) \times (C_S/C_U) \times 100$$

r_U = peak response of maleic acid from the *Sample solution*

r_S = peak response of maleic acid from the *Standard solution*

C_S = concentration of USP Maleic Acid RS in the *Standard solution* (mg/mL)

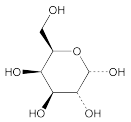
C_U = nominal concentration of the *Sample solution* (mg/mL)

Acceptance criteria: NMT 0.1%

SPECIFIC TESTS
- **WATER DETERMINATION,** *Method I* ⟨**921**⟩**:** 0.5%

ADDITIONAL REQUIREMENTS
- **PACKAGING AND STORAGE:** Preserve in well-closed containers.
- **USP REFERENCE STANDARDS** ⟨**11**⟩
  USP Fumaric Acid RS
  USP Maleic Acid RS

Galactose



$C_6H_{12}O_6$ 180.16
α-D-Galactopyranose [3646-73-9].

» Galactose is one of the products of the metabolism of lactose, a naturally occurring sugar in dairy products, by the digestive enzyme lactase.

Packaging and storage—Preserve in a tight container. No storage requirements specified.

USP Reference standards ⟨11⟩**—**
USP Dextrose RS
USP Galactose RS
USP Lactose Monohydrate RS

Appearance of solution—Dissolve, with heating at 50°, 10.0 g of Galactose in 50 mL of carbon dioxide-free water. The solution is not more intensely colored than a solution prepared immediately before use by mixing 3.0 mL of ferric chloride CS, 3.0 mL of cobaltous chloride CS, and 2.4 mL of cupric sulfate CS with dilute hydrochloric acid (10 g per L) to make 10 mL, and diluting 1.5 mL of this solution with the dilute hydrochloric acid to 100 mL. Make the comparison by viewing the substance and the solution downward in matched color-comparison tubes against a white surface (see *Color and Achromicity* ⟨631⟩).

Identification—

A: *Infrared Absorption* ⟨197K⟩.

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

Test solution—Dissolve 10 mg in 20 mL of a mixture of methanol and water (3:2).

Standard solution 1: 500 µg per mL in a mixture of methanol and water (3:2).

Standard solution 2—Prepare a solution using USP Galactose RS, USP Dextrose RS, and USP Lactose Monohydrate RS, each at a concentration of 500 µg per mL in a mixture of methanol and water (3:2).

Application volume: 2 µL.

Developing solvent system: propanol and water (85:15).

Procedure—Develop the plate in an unsaturated tank. After the solvent front has moved over 15 cm, remove the plate from the tank. Dry the plate with warm air, then spray the plate with a thymol solution (0.5 g in a mixture of alcohol and sulfuric acid [95:5]). Heat for 10 minutes in an oven at 130°. The R_F of the principal spot obtained from the *Test solution* corresponds to that obtained from the *Standard solution.* [NOTE—There must be three clearly resolved spots in the chromatogram for *Standard solution 2* in order for the results to be valid.]

Specific rotation ⟨781S⟩**:** between +78.0° and +81.5° at 20°.

Test solution: Transfer 10.0 g to a 100-mL volumetric flask, and dissolve in 80 mL of water. Add 0.2 mL of ammonia TS, allow to stand for 30 minutes, then dilute with water to volume.

Microbial enumeration tests ⟨61⟩ **and Tests for specified microorganisms** ⟨62⟩**—**It meets the requirements of the test for absence of *Salmonella* species, *Escherichia coli, Staphylcoccus aureus,* and *Pseudomonas aeruginosa.* The total aerobic microbial count does not exceed 1000 cfu per g, and the total combined molds and yeasts count does not exceed 100 cfu per g.

Acidity—Dissolve 10.0 g, with heating at 50°, in 40 mL of carbon dioxide-free water. Dilute with carbon dioxide-free water to 50 mL [NOTE—Use this solution for the *Barium* test]. To 30 mL of this solution, add 0.3 mL of phenolphthalein TS, and titrate with 0.01 N sodium hydroxide to a pink color: not more than 1.5 mL of 0.01 N sodium hydroxide is required to produce a pink color.

Water, *Method I* ⟨921⟩**:** not more than 1.0%.

Residue on ignition ⟨281⟩**:** not more than 0.1%.

Barium—

Standard solution—Add 6 mL of water to 5 mL of the solution prepared for the *Acidity* test.

Test solution—Add 5 mL of water and 1 mL of dilute sulfuric acid to 5 mL of the solution prepared for the *Acidity* test. Allow to stand for 1 hour: any opalescence in the *Test solution* is not more intense than that of the *Standard solution.*

Limit of lead—

Diluent—Dilute 12 mL of acetic acid with water to 100 mL. Mix equal parts of this solution and water to prepare the *Diluent.*

Lead standard stock solution—Transfer an accurately weighed quantity of lead nitrate, about 400 mg, to a 250-mL volumetric flask, dilute with water to volume, and mix.

Lead standard solution—Dilute 1.0 mL of *Lead standard stock solution* with water to 10 mL. Dilute 1.0 mL of this solution with water to 10 mL.

Standard solutions—To three identical flasks, add 0.5 mL, 1.0 mL, and 1.5 mL of *Lead standard solution,* respectively, and then add to each flask 20.0 g of galactose. Dilute with *Diluent* to 100 mL. To each flask add 2.0 mL of ammonium pyrrolidinedithiocarbamate solution (10 g per L) and 10.0 mL of methyl isobutyl ketone, then shake for 30 seconds. [NOTE—Protect from light.] Allow the layers to separate, and use the methyl isobutyl ketone (upper) layer for analysis.

Test solution—Dissolve 20.0 g of Galactose in *Diluent,* and dilute with *Diluent* to 100 mL. Add 2.0 mL of ammonium pyrrolidinedithiocarbamate solution (10 g per L) and 10.0 mL of methyl isobutyl ketone, and shake for 30 seconds. [NOTE—Protect from light.] Allow the layers to separate, and use the methyl isobutyl ketone (upper) layer for analysis.

Procedure—Concomitantly determine, at least in triplicate, the absorbances of the *Standard solutions* and the *Test solution* at 283.3 nm with a suitable atomic absorption spectrophotometer (see *Spectrophotometry and Light-Scattering* ⟨851⟩) equipped with a lead hollow-cathode lamp as the radiation source and an air–acetylene flame. Record the average steady readings for each of the *Standard solutions* and the *Test solution.* Plot the absorbances of the *Standard solutions* and the *Test solution* versus the amount of lead added. Extrapolate the line joining the points on the graph until it meets the concentration axis. The distance between this point and the intersection of the axes represents the concentration of lead in the *Test solution:* not more than 0.5 µg per g is found.

Galageenan

DEFINITION
Galageenan is the hydrocolloid obtained by extraction with water or aqueous alkali from the red seaweed class Rhodophyceae species *Eucheuma gelatinae.* Galageenan consists chiefly of potassium, sodium, calcium, magnesium, and am-