Amendment to the Enforcement Ordinance of the Food Sanitation Law and the Standards and Specifications for Foods and Food Additives

The government of Japan will designate peracetic acid,1-hydroxyethylidene-1,1-diphosphonic acid, and octanoic acid as food additives toenable the use of peracetic acid composition and establish specifications and standards for peracetic acid composition.

Summary

Under Article 10 of the Food Sanitation Law (hereinafter referred to as the "Law"), food additives shall not be used or marketed without authorization by the Minister of Health, Labour and Welfare (hereinafter referred to as "the Minister"). In addition, when specifications or standards are established for food additives based on Article 11 of the Law and stipulated in the Ministry of Health, Labour and Welfare Notification (Ministry of Health and Welfare Notification No. 370, 1959), those additives shall not be used or marketed unless they meet the standards or specifications.

In response to a request from the Minister, the Committee on Food Additives of the Food Sanitation Council that is established under the Pharmaceutical Affairs and Food Sanitation Council has discussed whether it is adequate to designate peracetic acid (PAA), 1-hydroxyethylidene-1,1-diphosphonic acid(HEDP), and octanoic acid as additives for the purpose of using them as ingredients of peracetic acid composition. The conclusion of the committee is outlined below.

Outline of conclusion

The Minister, based on Article 10 of the act, should designate PAA, HEDP, and octanoic acid as additives unlikely to harm human health and establish specifications and standards for these additives and peracetic acid composition based on Article 11.

Attachment

Peracetic Acid Composition, Peracetic Acid, 1-Hydroxyethylidene-1,1-diphosphonic Acid, Octanoic Acid

Standard for use

Peracetic acid composition: Permitted only for the surface disinfection of edible meat (beef, pork, and poultry), fruits, and vegetables. The maximum use level is 2.0 g for poultry, 1.80 g for beef and pork, 0.080 g for fruits and vegetables, as the amount of peracetic acid in 1 kg of a dipping or spray solution; and 0.136 g for poultry, 0.024 g for beef and pork, and 0.0048 g for fruits and vegetables, as the amount of 1-hydroxyethylidene-1,1-diphosphonic acid in 1 kg of a spray or dipping solution.

—*Fruits and vegetables include fresh vegetables and fruits that have been processed with simple processing, like peeling, cutting, and freezing.*

—*Edible meat includes dressed, cut, sliced, and ground meat.*

Peracetic acid: Permitted only as an ingredient of peracetic acid composition.

1-Hydroxyethylidene-1,1-diphosphonic acid: Permitted only as an ingredient of peracetic acid composition.

Octanoic acid: Permitted only for the purpose of flavoring foods or as an ingredient of peracetic acid composition.

Note: The use standard is established for hydrogen peroxide. Hydrogen peroxide shall be decomposed or removed before the completion of the final product. No use standard is established for acetic acid.

Standards for manufacturing

Peracetic acid: It shall be made of acetic acid and hydrogen peroxide that meet the corresponding existing specification.

Peracetic acid composition: It shall be a product manufactured by mixing 1-hydroxyethylidene-1,1-diphosphonic acid with peracetic acid or with acetic acid and hydrogen peroxide, or a product manufactured by adding octanoic acid to the above

mixture. Acetic acid, hydrogen peroxide, 1-hydroxyethylidene-1,1-diphosphonic acid, and octanoic acid that are used as ingredients of peracetic acid composition shall meet the existing specifications.

Peracetic Acid Composition

過酢酸製剤

Compositional specifications

Substance name Peracetic Acid Composition

[CAS number] [79-21-0, Peracetic acid]

Definition Peracetic Acid Composition is an aqueous solution containing Peracetic Acid, "Acetic Acid," "Hydrogen Peroxide," and "1-Hydroxyethylidene-1,1-diphosphonic Acid" or an aqueous solution containing these four substances and "Octanoic Acid." Peroctanoic acid may be produced from "Octanoic Acid" contained in "Peracetic Acid Composition."

Content Peracetic Acid Composition contains 12–15% of peracetic acid, 30–50% of acetic acid, 4–12% of hydrogen peroxide, and less than 1% of 1-hydroxyethylidene-1,1-diphosphonic acid. In addition, it can contain not more than 10% of actanoic acid.

Description Peracetic Acid Composition is a colorless, transparent liquid having a characteristic, pungent odor.

Assay

(1) Peracetic acid and acetic acid

Sample Solution Weigh accurately about 1 g of Peracetic Acid Composition, and add water to make exactly 100 ml.

Procedure Pour 5 ml of ethanol, then 10 ml of water into an octadecylsilanized silica gel minicolumn (500 mg), and discard the effluent. Pour 10 ml of the sample solution into the column, and collect the effluent in a 100-ml beaker. Pour 10 ml of water into the column, add the effluent to the beaker, and add about 50 ml of water to the beaker. Titrate the resulting solution with 0.1 mol/L sodium hydroxide using a potentiometer. Record the amounts (a ml and b ml) of the sodium hydroxide solution consumed at the first and second inflection points to determine each content.

$$\text{Content (\%) of peracetic acid } (C_2H_4O_3) = \frac{(b-a)\times 0.1\times 76.05}{\text{Weight (g) of the sample}}$$

$$\text{Content (\%) of acetic acid } (C_2H_4O_2) = \frac{a\times 0.1\times 60.05}{\text{Weight (g) of the sample}}$$

(2) Hydrogen peroxide

Test Solution Weigh accurately about 1 g of Peracetic Acid Composition, and add water to make exactly 100 ml. Transfer 10 ml of this solution, exactly measured, to a 250-ml Erlenmeyer flask, and add 75 ml of sulfuric acid TS (0.5 mol/L), cooled with ice.

Procedure Add 2 drops of ferroin TS to the test solution, and titrate with 0.1 mol/L cerium(IV) sulfate. The endpoint is when the color of the solution changes from orange to colorless through light red. Determine the content by the formula:

$$\text{Content (\%) of hydrogen peroxide } (H_2O_2) = \frac{\text{Consumption (ml) of 0.1 mol/L cerium(IV) sulfate} \times 0.1 \times 17.00}{\text{Weight (g) of the sample}}$$

(3) 1-Hydroxyethylidene-1,1-diphosphonic acid

Test Solution Weigh accurately about 0.2 g of Peracetic Acid Composition, and add water to make 50 ml. Transfer 3 ml of this solution, exactly measured, to a 100-ml beaker, and add 50 ml of water. To this solution, add 1 drop of phenolphthalein TS. When the solution turns light red, add sulfuric acid TS (2.5 mol/L) until the light red color disappears. To this solution, add 2 ml of sulfuric acid TS (2.5 mol/L), and stir. Then add 0.4 g of ammonium peroxodisulfate, and stir. Heat the resulting solution with boiling chips on a hot plate for 90 minutes while replenishing the lost water, and then continue to heat until the volume of the solution is reduced to about 10 ml. After cooling, add 2 drops of phenolphthalein TS, then add 1 mol/L sodium hydroxide TS until the color of the solution becomes faint red. Transfer this solution to a 50-ml volumetric flask, wash the boiling chips and the beaker a few times with a small amount of water, and add the washings to the flask. Make up with water to 50 mL and refer to the resulting solution as the sample solution. Measure exactly 10 ml of the sample solution, add 2.0 ml of antimony tartrate–molybdic acid TS, shake well, and allow to stand for 20 minutes.

Reference Solution Using 10 ml of water instead of the sample solution, proceed as directed for the sample solution, beginning with "add 2.0 ml of antimony tartrate–molybdic acid TS."

Standard Solutions Dissolve 0.2195 g of potassium dihydrogen phosphate in water to make exactly 1000 ml. To 5 ml of this solution, exactly measured, add water to make exactly 1000 ml. Use the resulting solution as the standard stock solution. Measure

exactly 0 ml, 3 ml, 5 ml, 10 ml, 15 ml, and 20 ml of the standard stock solution in separate volumetric flasks, and add water to each solution to make exactly 50 ml of each. Measure exactly 10 ml each of the resulting solutions, and proceed as directed for the sample solution, beginning with "add 2.0 ml of antimony tartrate–molybdic acid TS".

Procedure Measure the absorbance of the test solution and the standard solutions at 650 nm to prepare a calibrate curve. Determine the concentration of phosphorus in the test solution from the calibration curve and the absorbance of the test solution, and calculate the content of 1-hydroxyethylidene-1,1-diphosphonic acid ($C_2H_8O_7P_2$) by the formula:

$$\text{Content (\%)of } (C_2H_8O_7P_2) = \frac{\text{Concentration (μg /ml) of phosphorus} \times 206.0}{\text{Weight (g) of the sample} \times 61.94 \times 12}$$

(4) Octanoic acid

Test Solution Weigh accurately about 0.7 g of Peracetic Acid Composition, add a 1:1 mixture of water and acetonitrile to make 50 ml. To 5 ml of this solution, exactly measured, add a 1:1 mixture of water and acetonitrile to make exactly 20 ml.

Standard Solutions Weigh accurately about 0.2 g of octanoic acid for assy, add a 1:1 mixture of water and acetonitrile to make 100 ml. Use this solution as the standard stock solution. Measure exactly 0.5 ml, 1 ml, 2.5 ml, 5 ml, and 10 ml of the standard stock solution in separate volumetric flasks, and add a 1:1 mixture of water and acetonitrile to each solution to make exactly 20 ml of each.

Procedure Analyze 20 μl portions of the test solution and the standard solutions by liquid chromatography using the operating condition given below. Measure the peak areas of octanoic acid in the standard solutions to prepare a calibration curve. Determine the concentration (μg/ml) of octanoic acid in the test solution from the calibration curve and the peak area of actanoic acid in the test solution, and calculate the content of octanoic acid ($C_8H_{16}O_2$) by the formula:

$$\text{Content (\%) of octanoic acid } (C_8H_{16}O_2) = \frac{\text{Concentration (μg ml) of octanoic acid in the test solution}}{\text{Weight (g) of the smple} \times 50}$$

Operating Conditions

Detector: Ultraviolet absorption spectrophotometer (determination wavelength 210 nm)

Column: A stainless steel tube of 4.6 mm internal diameter and 25 cm length.

Column packing material: 5-μm octadecylsilanized silica gel for liquid chromatography.

Column temperature: 30°C.

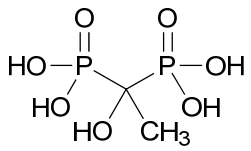
Mobile phase: A solution prepared by dissolving 0.12 g of acetic acid in 350 ml of

water and adding 650 ml of acetonitrile.

Flow rate: 1.0 ml/minute.

1-Hydroxyethylidene-1,1-diphosphonic Acid

1-ヒドロキシエチリデン-1,1-ジホスホン酸



Compositional specifications

Substance name 1-Hydroxyethylidene-1,1-diphosphonic Acid

Molecular formula $C_2H_8O_7P_2$

Molecular weight 206.03

Chemical name [CAS number]

(1-Hydroxyethane-1,1-diyl)diphosphonic acid [2809-21-4]

Content 1-Hydroxyethylidene-1,1-diphosphonic Acid contains 58.0–62.0% of 1-hydroxyethylidene-1,1-diphosphonic acid ($C_2H_8O_7P_2$).

Description 1-Hydroxyethylidene-1,1-diphosphonic Acid is a colorless to light yellow, clear liquid.

Purity

(1) Specific gravity 1.430–1.471.

(2) pH Not more than 2.0 (1.0 g, water 100 ml).

(3) Chlorides Not more than 0.004% as Cl.

Weigh accurately about 25 g of 1-Hydroxyethylidene-1,1-diphosphonic Acid, add about 50 ml of water and 3 ml of nitric acid. Titrate the resulting solution with 0.005 mol/L silver nitrate using a potentiometer. Record the amount (a ml) of the silver nitrate solution consumed at the endpoint to determine the amount of chlorides by the following formula. If more than one inflection point exists, the endpoint is the last

inflection point.

$$\text{Amount (\%)of chlorides (Cl)} = \frac{a \times 0.005 \times 3.545}{\text{Weight (g) of the sample}}$$

(4) Phosphorous acid Not more than 4.0% as H_3PO_3.

Weigh accurately about 1.5 g of 1-Hydroxyethylidene-1,1-diphosphonic Acid in an iodine flask, add 20 ml of water and 50 ml of phosphate buffer (pH 7.3), and adjust the pH of the mixture to 7.3 with sodium hydroxide solution (1 in 2). Add 25 ml of 0.05 mol/L iodine, exactly measured, immediately stopper tightly, and allow to stand for 15 minutes at a dark place. Add 5 ml of acetic acid, and titrate the excess iodine with 0.1 mol/L sodium thiosulfate (indicator: 1–3 ml of starch TS). Add starch TS near the endpoint, when the color of solution changes to light yellow. The endpoint is when the blue color produced disappears. Separately, perform a blank test.

Each ml of 0.05 mol/L iodine = 4.10 mg of H_3PO_3.

(5) Lead Not more than 5.0 μg/g as Pb.

Sample Solution Weigh 0.80 g of 1-Hydroxyethylidene-1,1-diphosphonic Acid in a platinum, quartz, or porcelain crucible or a quartz beaker. Add 1 ml of sulfuric acid, heat by gradually increasing the temperature until the sample is charred and white fumes of sulfuric acid are no longer evolved. If necessary, add sulfuric acid again and heat until the sample is almost charred. Cover the container loosely with a lid if necessary, heat in an electric furnace by gradually increasing the temperature, and ignite at 450–600°C to incinerate the sample. When a charred mass remains, crush it with a glass rod if necessary, moisten with 1 ml of diluted sulfuric acid (1 in 4) and 1 ml of nitric acid, heat until white fumes of sulfuric acid are no longer evolved, and ignite in the electric furnace to completely incinerate the sample. To the residue, add 10 ml of diluted hydrochloric acid (1 in 4), heat on a water bath, and evaporate to dryness. To the residue, add 20 ml of diluted hydrochloric acid (1 in 4), boil with a watch glass covering the container for 5 minutes, and cool.

Test Solution To the sample solution, add 10 ml of diammonium hydrogen citrate solution (1 in 2), and then add ammonium solution until the color of the solution changes from yellow to light yellow-green using 1 ml of thymol blue TS as the indicator. Transfer this solution to a separating funnel or centrifuge tube. Wash the container used for incineration with a small amount of water or warm water, and add the washings to the funnel or tube, whichever may be appropriate. Add 5 ml of ammonium pyrrolidine dithiocarbamate solution (3 in 100), and allow to stand for 5 minutes. Add exactly 10 ml of butyl acetate, shake for 5 minutes, and allow to stand or centrifuge. Collect the butyl acetate layer.

Control solution Measure exactly 1 ml of Lead Standard Stock Solution, and add water to make exactly 100 ml. Measure exactly 4 ml of Lead Standard Solution, and proceed as directed for the test solution.

Procedure Conduct tests for the test and control solutions by Method 1.

(6) Iron Not more than 10 μg/g as Fe.

Sample Solution Weigh accurately about 0.2 g of 1-Hydroxyethylidene-1,1-diphosphonic Acid in an appropriate container, and add 5 ml of nitric acid. Incinerate the sample in a microwave digestion equipment at 230°C. After cooling, transfer the residue in a 50-ml volumetric flask, and make up with water to volume.

Test Solution and Standard Solutions To an appropriate amount of Iron Standard Solution, exactly measured, add diluted nitric acid (1 in 10) to prepare standard stock solutions containing iron (Fe = 55.85) at the concentrations of 10, 25, 50, 100, and 200 ng per ml. Measure 10 ml each of the sample solution and the standard stock solutions in separate flasks, and add 40 μl of the internal standard to each flask to prepare the test solution and the standard solutions, respectively. Prepare the internal standard as follows: To 1.0 ml of Yttrium Standard Stock Solution, add diluted nitric acid (1 in 10) to make 100 ml.

Procedure Proceed as directed in the Internal Standard Method under Inductively Coupled Plasma-Atomic Emission Spectrometry to measure emissions of the test solution and the standard solutions, and prepare a calibration curve. Determine the concentration (ng/ml) of iron in the test solution from the calibration curve, and calculate the iron amount (μg/g) by the formula:

$$\text{Amount (μg/g) of iron (Fe)} = \frac{\text{Iron concentration (ng/ml) in the test solution}}{\text{Weight (g) of the sample} \times 20}$$

(7) Arsenic Not more than 6.7 μg/g as As_2O_3 (0.30 g, Method 1, Apparatus B).

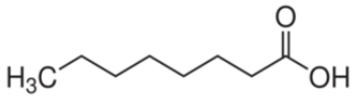
Assay Dissolve about 3 g of 1-Hydroxyethylidene-1,1-diphosphonic Acid, accurately weighed, in 150 ml of water. Titrate this solution with 1 mol/L sodium hydroxide using a potentiometer while stirring. The endpoint is the second inflection point. Record the consumption (a ml) of the sodium hydroxide at the endpoint, and calculate the content of 1-hydroxyethylidene-1,1-diphosphonic acid ($C_2H_8O_7P_2$) by the formula:

$$\text{Content (\%) of } C_2H_8O_7P_2 = \frac{a \times 206.0}{\text{Weight (g) of the sample} \times 30} - \text{amount (\%) of phosphorous acid} \times 1.675$$

Octanoic Acid

Caprylic Acid

オクタン酸



Compositional specifications

Substance name Octanoic Acid

Molecular formula $C_8H_{16}O_2$

Molecular weight 144.21

Chemical name [CAS number] Octanoic acid [124-07-2]

Content Octanoic Acid contains not less than 95.0% of octanoic acid ($C_8H_{16}O_2$).

Description Octanoic Acid is a colorless, oil-like liquid. It has a faint odor.

Identification Determine the absorption spectrum of Octanoic Acid as directed in the Liquid Film Method under Infrared Spectrophotometry, and compare with the Reference Spectrum. Both spectra exhibit absorptions having almost the same intensity at the same wavenumbers.

Purity

(1) Acid value 366–396.

Weigh accurately about 0.3 of Octanoic Acid, and proceed as directed in Acid Value under the Flavoring Substances Tests.

(2) Lead Not more than 2.0 μg/g as Pb.

Test Solution Weigh 2.0 g of Octanoic Acid into a platinum, quartz, or porcelain crucible or a quartz beaker. Add 1 ml of sulfuric acid, and heat by gradually increasing the temperature until the sample is charred and white fumes of sulfuric acid are no longer evolved. If necessary, add sulfuric acid again, and heat until the sample is almost charred. Cover the container loosely with a lid, heat it in an electric furnace by

gradually increasing the temperature, and ignite at 450–600°C until the sample is incinerated. When a chatted mass remains, crush with a glass rod if necessary, moisten it with 1 ml of diluted sulfuric acid (1 in 4) and 1 ml of nitric acid, heat until white fumes are no longer evolved, and ignite in the electric furnace to completely incinerate the sample. To the residue, add 10 ml of diluted hydrochloric acid (1 in 4), heat on a water bath, and evaporate to dryness. To the residue, add a small amount of diluted nitric acid (1 in 100), and dissolve it by warming. After cooling, make up with diluted nitric acid (1 in 100) to exactly 10 ml. A heat-resistant glass beaker can be used when the incineration procedure is performed at a temperature not more than 500°C.

Control Solution Measure exactly 1 ml of Lead Standard Stock Solution, and add water to make exactly 100 ml. To 4 ml of this solution, exactly measured, add diluted nitric acid (1 in 100) to make exactly 10 ml.

Procedure Proceed as directed in Method 1 under the Lead Limit Test.

(3) Decanoic acid Not more than 3.0%.

Test Solution Use Octanoic Acid as the sample.

Control Solution Weigh 0.3 ml of decanoic acid and add 10 ml of Octanoic Acid as the sample.

Procedure Analyze the test solution and the control solution by gas chromatography using the operating conditions specified in Assay to confirm decanoic acid of the test solution. Determine the sum (A_T) of the peak areas of all the peaks that appear within the specified measurement time and the peak area (A_S) of decanoic acid. Calculate the amount of decanoic acid by the formula.

$$\text{Amount (\%) of decanoic acid} = \frac{A_S}{A_T} \times 100$$

Water Content Not more than 0.4% (5 g, Direct Titration).

Residue on Ignition Not more than 0.1% (10 g, 800°C, 15 minutes).

Assay Proceed as directed under the Peak Area Percentage Method in the Gas Chromatographic Assay of Flavoring Agents under the Flavoring Substances Tests. Use operation conditions (1). Use a silicate glass capillary column (0.25–0.53 mm in internal diameter and 30–60 m in length) coated with a 0.25- to 1-μm thick layer of polyethylene glycol for gas chromatography. The column temperature should be raised from 150 to 230°C at a rate of 5°C/minute and then maintained at 230°C for 24 minutes.

Regents and Solutions

Antimonyl Potassium Tartarate TS Dissolve 1.37 g of bis[(+)-tartrato]diantimonate(III)dipotassium trihydrate by adding 350 ml of water gradually, and make up with water to 500 ml.

Antimony Tartrate–Molybdic Acid TS To 50 ml of sulfuric acid TS (2.5 mol/L), add 5 ml of antimonyl potassium tartarate TS, 15 ml of a solution of hexaammonium heptamolybdate tetrahydrate (1 in 25), and 30 ml of ascorbic acid TS, and stir well. Prepare fresh before use.

Ascorbic Acid TS Dissolve 1.76 g of L-ascorbic acid in water to make 100 ml.

Bis[(+)-tartrato]diantimonate(III) Dipotassium Trihydrate $C_8H_4K_2O_{12}Sb_2 \cdot 3H_2O$ [K 8533]

Cerium(IV) Sulfate Tetrahydrate $Ce(SO_4)_2 \cdot 4H_2O$ [K8976]

Ferroin TS To 0.70 g of iron(II) sulfate heptahydrate, add 70 ml of water and 1.78 g of **1,10-phenanthroline chloride** monohydrate to dissolve, and make up with water to 100 ml.

Decanoic Acid $C_{10}H_{20}O_2$ A colorless to light yellow, clear liquid or white to faint yellow crystals or lumps.

Content Not less than 99.0%.

Identification Proceed as directed in the Potassium Bromide Disc Method under Infrared Spectrophotometry. It exhibits absorption bands at about 2676 cm^{-1}, 1700 cm^{-1}, 1299 cm^{-1}, 1268 cm^{-1}, 1232 cm^{-1}, 1200 cm^{-1}, 1075 cm^{-1}, 934 cm^{-1}, 825 cm^{-1}, and 686 cm^{-1}.

Congealing point 29–33°C.

Assay Weigh accurately about 0.05 g of Octanoic Acid for Assay, add 1 ml of *N,O*-bis(trimethylsilyl)trifluoroacetamide, stopper tightly, and mix well. Heat the mixture on a water bath for 30 minutes, and cool to room temperature. Analyze appropriate portions of the resulting solution by gas chromatography using the operating conditions given below. Determine the area percentage of the main peak.

Operating conditions

Detector: Flame-ionization detector.

Column: A silicate glass capillary column (0.53 mm in internal diameter and 15 m in length) coated with a 1.5-µm thick layer of dimethylpolysiloxane for gas chromatography.

Column temperature: Raise the temperature at a rate of 10°C/minutes from 60°C to 280°C.

Injection port temperature: 280°C.

Detector temperature: 280°C.

Injection method: Split (20:1). Set conditions so that any component of the sample does not exceed the column acceptable range.

Carrier gas: Helium.

Flow rate: Adjust so that the peaks of components to be determined appear in 5–20 minutes of injection.

Octadecylsilanized Silica Gel Minicolumn (500 mg) Use a polyethylene column (10–25 mm in internal diameter) packed with 0.5 g of octadecylsilanized silica gel or a column comparable in separation property to the former one.

Octanoic Acid for Assay $CH_3(CH_2)_6COOH$ A colorless to light yellow, clear liquid.

Content Not less than 98.0%.

Identification Proceed as directed in the Liquid Film Method under Infrared Spectrophotometry. It exhibits absorption bands at about 2930 cm^{-1}, 2860 cm^{-1}, 1710 cm^{-1}, 1460 cm^{-1}, 1420 cm^{-1}, 1280 cm^{-1}, 1230 cm^{-1}, 1200 cm^{-1}, 1110 cm^{-1}, 940 cm^{-1}, and 720 cm^{-1}.

Purity (1) Congealing point 15–17°C.

(2) Refractive index n_d^{20}: 1.425–1.431.

(3) Specific gravity d_{20}^{20}: 0.909–0.915.

Assay Weigh accurately about 0.05 g of Octanoic Acid for Assay, add 1 ml of *N,O*-bis(trimethylsilyl)trifluoroacetamide, stopper tightly, and mix well. Heat the mixture on a water bath for 30 minutes, and allow to cool. Analyze appropriate portions of the resulting solution by gas chromatography using the operating conditions given below. Determine the area percentage of the main peak.

Operating conditions

Detector: Flame-ionization detector.

Column: A silicate glass capillary (0.53 mm in internal diameter and 15 m in length) coated with a 1.5-µm thick layer of dimethylpolysiloxane for gas chromatography.

Column temperature: Raise the temperature at a rate of 10°C/minutes from 50°C to 280°C, and then maintain at 280°C for 2 minutes.

Injection port temperature: 280°C.

Detector temperature: 280°C.

Injection method: Split (20:1). Set conditions so that any component of the sample does not exceed the column acceptable range.

Carrier gas: Helium.

Flow rate: Adjust so that the peaks of components to be determined appear in 5–20 minutes of injection.

1,10-Phenanthroline Chloride Monohydrate $C_{12}H_9ClN_2 \cdot H_2O$ [K8202]

Phosphate Buffer (pH 7.3) Dissolve 138 g of monosodium phosphate in 800 ml of water. Adjust the pH of this solution to 7.3 with sodium hydroxide solution (1 in 2), and add water to make 1000 ml.

Sulfuric Acid TS (0.5 mol/L) Add 14 ml of sulfuric acid gradually to 350 ml of water. After cooling, make up with water to 500 ml.

Sulfuric Acid TS (2.5 mol/L) Add 70 ml of sulfuric acid gradually to 350 ml of water. After cooling, make up with water to 500 ml.

Volumetric Solution

0.1 mol/L Cerium(IV) Sulfate This solution contains 40.43 g of cerium(IV) sulfate tetrahydrate ($Ce(SO_4)_2 \cdot 4H_2O$, molecular weight: 404.30) per 1000 ml.

To about 40.4 g of cerium(IV) sulfate tetrahydrate, add 50 ml of sulfuric acid, and mix. To the mixture, add 900 ml of water in 20-ml portions while stirring, being careful about heat generation. Allow to stand for 24 hours, filter the solution through a glass filter, and add water to make 1000 ml.

Standardization Measure exactly 25 ml of 0.1 mol/L cerium(IV) sulfate, add 30 ml of diluted sulfuric acid (1 in 6), and titrate with 0.1 mol/L ammonium ferrous sulfate (indicator: about 0.2 ml of ferroin TS). The endpoint is when the color of the solution changes blue-green to yellow-red. Calculate the normality factor by the formula:

$f = f_1 \times V/25$

f : factor of 0.1 mol/L cerium(IV) sulfate

f_1 : factor of 0.1 mol/L ammonium ferrous sulfate

V : volume (ml) of 0.1 mol/L ammonium ferrous sulfate consumed

0.005mol/L Silver Nitrate This solution contains 0.8493 g of silver nitrate ($AgNO_3$, molecular weight:169.87) per 1,000 ml. Prepare by diluting 0.1 mol/L silver nitrate with water to 20 times its original volume.

Standard Solutions

Yttrium Standard Stock Solution Each ml of this solution contains 1 mg of yttrium (Y). Use a product prepared for inductively coupled plasma-atomic emission spectrometry.

Infrared Reference Spectrum

Octanoic Acid