

1. Reagents and Test Solutions

Absolute Ethanol See Ethanol, Absolute

Absorbent Cotton Use absorbent cotton specified under the Japanese Pharmacopoeia.

Acetaldehyde CH_3CHO (Extra grade)

Acetate Buffer Weigh 82 g of anhydrous sodium acetate, and dissolve in 140 ml of water. Add 25 ml of acetic acid and water to make 250 ml. Adjust the pH to 5.51 \pm 0.03 with acetic acid or sodium acetate solution (2 15).

Acetate Buffer (pH 5.4)

Solution 1: To 5.78 ml of acetic acid, add water to make 1,000ml.

Solution 2: Weigh 8.5 g of anhydrous sodium acetate, and dissolve in water to make 1,000 ml.

Mix 176 parts of solution 1 and 824 parts of solution 2. Adjust the pH to 5.4 with either of the solutions 1 and 2.

Acetic Acid CH_3COOH (Guaranteed)

Acetic Acid for Nonaqueous Titration Measure 1,000 ml of acetic acid, add 5 g of chromium trioxide, and allow to stand overnight. Filter, and distill the filtrate. To the distillate obtained at 115 or above, add 20 g of acetic anhydride, and redistill. Use the fraction obtained at a constant boiling temperature 117 - 118 .

Acetic Acid, Dilute Weigh 6 g of acetic acid, and add water to make 100 ml (1 mol/l).

Acetic Acid - Sodium Acetate Buffer (pH 4.5) for Iron Limit Test Dissolve 75.4 ml of acetic acid and 111 g of sodium acetate in water to make 1,000 ml.

Acetic Anhydride $(\text{CH}_3\text{CO})_2\text{O}$ (Guaranteed)

Acetic Anhydride - Pyridine TS Weigh 25 g of acetic anhydride, and add dehydrated pyridine to make 100 ml. Prepare freshly before use.

Acetone $(\text{CH}_3\text{COCH}_3)$ (Guaranteed)

Acetonitrile CH_3CN (Guaranteed)

Acetyl Chloride for Linalool Assay CH_3COCl Measure 128 ml of acetic acid, transfer into a 300-ml three-necked flask, and equip the flask with a dropping funnel and a reflux condenser by ground joints. Cool the flask in an ice water bath. Add dropwise 100 g of phosphorus trichloride slowly, keeping the temperature at 10 or below, and allow to stand for 30 minutes. Boil for 30 minutes, allow to stand, and separate the content into two layers. Transfer the supernatant carefully into the distillation flask, add 5 ml of acetic acid, and distill it under Method 2 in Boiling Point and Amount of Distillate. To the fraction distilled at 45 or above, add 5 g of freshly molten anhydrous sodium acetate, distill again in the same manner, and collect the distillate at 50 or above. In these procedures, the lower end of the condenser is equipped with a three-branched adapter, connected with about 100-ml flasks to collect distillate and with calcium chloride tube at the branch of the adapter. All parts of the apparatus must be connected by ground-glass joints. Prepare freshly before use.

2-Acetyl 4-Tetrahydroxybutyl Imidazole $\text{C}_9\text{H}_{14}\text{N}_2\text{O}_5$ 2-Acetyl 4-Tetrahydroxy-butyl Imidazole occurs as a grayish-white crystal or crystalline powder, freely soluble in methanol and ethanol and sparingly soluble in water.

Melting point: 234 - 236

Purity: Dissolve 10.0 mg of 2-Acetyl 4-Tetrahydroxybutyl Imidazole into 100 ml

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of decarboxylated methanol. Proceed the Liquid Chromatography under the following conditions. Only one peak of 2-acetyl 4-tetrahydroxybutyl imidazole is observed.

Operating conditions

Detector: Detector for absorbances in the ultraviolet region (determination wavelength: 280 nm).

Packing material of column: 5- μ m octadecylsilanized silica gel.

Column: Stainless pipe 4.6 mm in internal diameter and 15 cm in length.

Mobile phase: Mixture of 0.2% w/v phosphoric acid and methanol (45 : 60).

Flow rate: 0.6 ml/min.

Acetylene See Dissolved Acetylene

Acidic Ferrous Sulfate TS See Ferrous Sulfate, Acidic.

Acidic Stannous Chloride TS See Stannous Chloride TS, Acidic.

Acriflavine Hydrochloride $C_{14}H_{14}ClN_3 \cdot HCl$ Acriflavine Hydrochloride occurs as a dark red - brown crystalline powder. Acriflavine Hydrochloride solution (1 : 100) is reddish brown. Measure 1 ml of this solution, and add 30 ml of water. The color of the solution changes to yellow, and a fluorescence is emitted. To the solution, add 1 ml of hydrochloric acid. The fluorescence disappears. To Acriflavine Hydrochloride solution (1 : 10), add sodium hydrogen carbonate solution (1 : 20). An effervescence occurs.

Acrylate Resin for Adsorption Porous resin made as adsorbent.

Active Carbon Use medicinal carbon in the Japanese Pharmacopoeia.

Agar (Agar, Guaranteed)

Albumin TS Take carefully egg white from one fresh egg, add 100 ml of water. Mix egg white and water while shaking sufficiently and filter. Prepare freshly before use.

Aldehyde-free Ethanol See Ethanol, Aldehyde - free.

Alizarin S $C_{14}H_5O_2(OH)_2SO_3Na \cdot H_2O$ [Alizarin Red S (Sodium Alizarinsulfonate), Guaranteed]

Alizarin Yellow GG $C_{13}H_8N_3NaO_5$ [Alizarin Yellow GG (Salicyl Yellow), Guaranteed]

Alizarin Yellow GG TS Weigh 0.1 g of alizarin yellow GG, and dissolve in 100 ml of ethanol. Filter if necessary.

Alizarin Yellow GG - Thymolphthalein TS Mix 10 ml of alizarin yellow GG TS and 20 ml of Thymolphthalein TS.

Alkaline Cupric Citrate TS See Cupric Citrate TS, Alkaline.

Alkaline Pyrogallol Solution See Pyrogallol Solution, Alkaline.

Alumina Alumina occurs as a white powder. It is almost odorless, tasteless, and insoluble in water and organic solvents.

Particle size: Alumina passes a 149- μ m standard sieve and hardly pass a 74- μ m sieve.

pH: Not more than 11.0. Weigh 50 g of Alumina, add 200 ml of water, and boil for 30 minutes. Cool, and filter. Measure the pH of the filtrate.

Adsorption: 0.1 - 0.2. Stuff glass wool into the end of glass tube (18 mm in

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internal diameter), transfer 30 g of Alumina into the tube, and stuff it while patting the tube until the height of the layer of alumina does not change. Cover the surface of alumina layer with the small, circular filter paper, and pour benzene to the surface of alumina to moisten completely. Pour immediately 20 ml of a solution of picric acid in benzene (1 : 20), and when the surface of the solution reaches the top of alumina layer, pour 20 ml of benzene again. Measure the height of alumina layer (L) and the height of the layer absorbing picric acid (l). The adsorption obtained by the following formula:

$$\text{Adsorption} = \frac{L}{l \times 30}$$

Aluminum Al (Guaranteed)

Aluminum Chloride $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$ [Aluminum Chloride (Hexahydrate) (Aluminum Chloride), Guaranteed]

Aluminium Potassium Sulfate $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ (Aluminum Potassium Sulfate, Dodecahydrate, Guaranteed)

4-Amino-benzen Sulfonic Acid $\text{C}_6\text{H}_7\text{NO}_3\text{S}$ 4-Amino-benzen Sulfonic Acid occurs as white to whitish powder.

Specific absorbance: $E_{1\text{ cm}}^{1\%}$ (absorption maximum near the 248 nm) = not less than 869

Weigh 10.0 mg of 4-Amino-benzen Sulfonic Acid, previously dried in a vacuum desiccator, add ammonium acetate solution (3 : 2,000) and dissolve to make exactly 100 ml. Use this solution as solution A. Exactly measure 10 ml of solution A, add ammonium acetate solution (3 : 2,000) to make exactly 100 ml. Measure the adsorption of this solution.

Purity: Other aromatic compounds Exactly measure 10 ml of solution A, add ammonium acetate solution (3 : 2,000) to make exactly 100 ml. Measure 20 μl of this solution, perform Liquid Chromatography using the operating conditions directed under Purity (6) for Food Yellow No.4 specified in the Monographs, JSFA- . Only one peak is observed.

4-Amino-5-methoxy-2-methylbenzen Sulfonic Acid $\text{C}_8\text{H}_{11}\text{NO}_4\text{S}$ 4-Amino-5-methoxy-2-methylbenzen Sulfonic Acid occurs as a whitish powder.

Specific absorbance: Weigh 10.0 mg of 4-Amino-5-methoxy-2-methyl-benzen Sulfonic Acid, previously dried for 24 hours in a desiccator under a reduced pressure, dissolve in ammonium acetate solution (3 : 2,000) to make exactly 100 ml, and use this solution as solution A. Measure exactly 10 ml of solution A, and add ammonium acetate solution (3 : 2,000) to make exactly 100 ml. The solution exhibits absorption maximums at each of the wavelengths of 218 nm, 250 nm, and 291 nm. The specific absorbance of 4-Amino-5-methoxy-2-methylbenzen Sulfonic Acid at 250 nm is not less than 362.

Purity: Other aromatic compounds Measure exactly 1.0 ml of solution A, and add ammonium acetate solution (7.7 : 1,000) to make exactly 100 ml.

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Measure 20 ml of this solution, and perform Liquid Chromatography under the operating conditions specified in Purity (8) for Food Red No. 40 specified in the Monographs, JSFA- . Only one peak of 4-amino-5-methoxy-2-methylbenzen sulfonic acid is observed.

1-Amino-2-naphthol-4-sulfonic Acid $C_{10}H_5(NH_2)(OH)SO_3H$ (Guaranteed)

1-Amino-2-naphthol-4-sulfonic Acid TS Weigh 0.2 g of 1-amino-2-naphthol-4-sulfonic acid, dissolve in 195 ml of sodium hydrogen sulfite solution (3 20) and 5 ml of anhydrous sodium sulfite solution (1 5), and filter if necessary. Stopper tightly, and store in a dark, cold place. Use within 10 days after preparation.

4-Aminoantipyrine $C_{11}H_{13}N_3O$ [4-Aminoantipyrine (4-Amino-1,5-dimethyl-2-phenyl-3H-pyrazole-3-one), Guaranteed]

Ammonia TS Measure 400 ml of aqueous ammonia, and add water to make 1,000 ml.

Ammonia - Ammonium Chloride Buffer(pH 10.7) Weigh 67.5 g of ammonium chloride, dissolve in 570 ml of aqueous ammonia, and add freshly boiled and cooled water to make 1,000 ml.

Ammonium Acetate CH_3COONH_4 (Guaranteed)

Ammonium Acetate Buffer Weigh 77 g of ammonium acetate, and dissolve in 10 ml of acetic acid and water to make 1,000 ml.

Ammonium Acetate Buffer (pH 3.0)

Solution 1: Dissolve 10 g of ammonium acetate to make 100 ml.

Solution 2: Add water to 31.0 g of acetic acid to make 100 ml.

Mix solution 1 and solution 2, and adjust pH 3.0 using both solution.

Ammonium Amidosulfate $NH_4OSO_2NH_2$ (Guaranteed)

Ammonium Carbonate (Guaranteed)

Ammonium Carbonate TS Weigh 20 g of ammonium carbonate, and dissolve in 20 ml of ammonia TS and water to make 100 ml.

Ammonium Cerium Nitrate $(NH_4)_2Ce(NO_3)_6$ [Ammonium Cerium () Nitrate, Guaranteed]

Ammonium Cerium() Sulfate $Ce(SO_4)_2 \cdot 2(NH_4)_2SO_4 \cdot 4H_2O$ [Ammonium Cerium() Sulfate (Tetrahydrate), Guaranteed]

Ammonium Chloride NH_4Cl (Guaranteed)

Ammonium Metavanadate NH_4VO_3 [Ammonium Vanadate (Ammonium Metavanadate), Guaranteed]

Ammonium Molybdate $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ (Guaranteed)

Ammonium Molybdate TS Weigh 6.5 g of powdered molybdenum trioxide, and dissolve in a mixture of 14 ml of water and 14.5 ml of aqueous ammonia. Cool, and add slowly the solution to a cooled mixture of 32 ml of nitric acid and 40 ml of water while stirring. Allow to stand for 48 hours, and filter through asbestos. This solution deteriorates upon long standing. Measure 5 ml of this solution, and add 2 ml of disodium phosphate solution (1 8). If an abundant and yellow precipitate is not formed immediately or after slight warming, the solution must not be used. Store protecting from light. If a precipitate is formed, use only the supernatant.

Ammonium Molybdate - Sulfuric Acid TS Weigh 18.8 g of ammonium molybdate,

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dissolve in 300 ml of water, add 150 ml of sulfuric acid, and add water to make 500 ml.

Ammonium Nickel Sulfate $\text{NiSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ [Ammonium Nickel () Sulfate Hexahydrate, (Guaranteed)]

Ammonium Nitrate NH_4NO_3 (Guaranteed)

Ammonium Oxalate $\text{H}_4\text{NOCCOONH}_4 \cdot \text{H}_2\text{O}$ [Ammonium Oxalate (Monohydrate), Guaranteed]

Ammonium Peroxo Disulfate $(\text{NH}_4)_2\text{S}_2\text{O}_8$ (Guaranteed)

Ammonium Persulfate See Ammonium Peroxo Disulfate.

Ammonium Pyrrolidine Dithiocarbamate $\text{C}_5\text{H}_{12}\text{N}_2\text{S}_2$ (Guaranteed)

Ammonium Sulfamate See Ammonium Amidesulfate.

Ammonium Sulfate $(\text{NH}_4)_2\text{SO}_4$ (Guaranteed)

Ammonium Sulfide TS $(\text{NH}_4)_2\text{S}$ [Ammonium Sulfide Solution (Colorless), Extra grade] Store in a small, completely filled light-resistant bottle.

Ammonium Tartrate $\text{H}_4\text{NOOCCH}(\text{OH})\text{CH}(\text{OH})\text{COONH}_4$ (Guaranteed)

Ammonium Thiocyanate NH_4SCN (Guaranteed)

Ammonium Thiocyanate - Cobalt Nitrate TS Weigh 17.4 g of ammonium thiocyanate and 2.8 g of cobalt nitrate, mix, and add water to make 100 ml.

Amyl Alcohol, Iso See 3-Methyl-1-butanol.

Amylase (Crystal) Amylase (Crystal) is α -amylase obtained from *Bacillus subtilis*. It occurs as a white odorless crystalline powder.

Weigh accurately about 1 g of starch, dry at 105 °C for 4 hours, and determine the loss on drying. Weigh the amount of starch equivalent to 2.0 g of dried starch, transfer into a Nessler tube, add 5 ml of phosphate buffer (pH 7) and water to make 50 ml. Heat in a water bath for 10 minutes while shaking occasionally, and allow to stand at 40 °C for 30 minutes. To this mixture, add 0.5 ml of Amylase (Crystal) solution (1 : 1,000), shake well, and allow to stand at 40 °C for 30 minutes. Add immediately 1 ml of sodium hydroxide solution (1 : 25), shake well, and cool. Add 2 drops of phenolphthalein TS, and turn upside down twice. A uniformly pink color develops.

Amylase TS Weigh 0.2 g of amylase (crystal), add 100 ml of water, shake well, and filter. Prepare freshly before use.

Anhydrous Cupric Sulfate See Cupric Sulfate, Anhydrous.

Anhydrous Disodium Phosphate See Disodium Phosphate, Anhydrous.

Anhydrous Disodium Phosphate for pH Determination See Disodium Phosphate, Anhydrous, for pH Determination.

Anhydrous Potassium Carbonate See Potassium Carbonate, Anhydrous.

Anhydrous Sodium Acetate See Sodium Acetate, Anhydrous.

Anhydrous Sodium Carbonate See Sodium Carbonate, Anhydrous.

Anhydrous Sodium Sulfate See Sodium Sulfate, Anhydrous.

Anhydrous Sodium Sulfite See Sodium Sulfite, Anhydrous.

Aniline $\text{C}_6\text{H}_5\text{NH}_2$ (Guaranteed)

Aniline Azo Schaeffer's Salt $\text{C}_{16}\text{H}_{11}\text{N}_2\text{NaO}_4\text{S}$ Aniline Azo Schaeffer's Salt is mono-sodium 6-hydroxy-5-(phenylazo)-2-naphthalene-6-sulfonate, and it occurs as an orange-red powder.

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Specific absorbance: $E_{1\text{ cm}}^{1\%}$ (absorption maximum near the 483 nm) = not less than 595

Weigh 10.0 mg of Aniline Azo Schaeffer's Salt, previously dried for 24 hours in a vacuum desiccator, add ammonium acetate solution (3 2,000) and dissolve to make exactly 100 ml. Use this solution as solution A. Exactly measure 10 ml of solution A, add ammonium acetate solution (3 2,000) to make exactly 100 ml. Measure the adsorption of this solution.

Purity: Other colors Exactly measure 10 ml of solution A, add ammonium acetate solution (3 2,000) to make exactly 100 ml. Measure 20 μl of this solution, perform Liquid Chromatography using the operating conditions directed under Purity (5) for Food Yellow No.5 specified in the Monographs, JSFA- . Only one peak is observed.

Anion-exchange Resin, Strongly Basic Strongly Basic Anion-exchange Resin is strongly basic quaternary ammonium salt of polystyrene and occurs as a yellow to yellow-brown powder. It passes a 590- μm standard sieve and hardly pass a 420- μm sieve.

Weigh about 50 g of Strongly Basic Anion-exchange Resin, immerse in water, allow to stand for 30 minutes, and pour the resin with water into a glass tube for chromatography (about 2.5 cm in internal diameter) to prepare the resin column. Pour 2,000 ml of sodium hydroxide solution (1 25) into the column, and flow at the rate of about 30 ml per minute. Then, pour water to wash the column until the washings show neutrality with phenolphthalein TS, and perform the following test:

Measure 10 ml of the resin, pour it with water into a glass tube for chromatography (1.5 cm in internal diameter), and flow 70 ml of 0.1 mol/l hydrochloric acid from the column at the rate of about 2 ml per minute. The pH of the outflow is 4.0 - 8.0.

Anion-exchange Resin, Weakly Basic Weakly Basic Anion-exchange Resin is weakly basic polystyrene polyamine and occurs as a yellow to yellow-brown powder. It passes a 590- μm standard sieve and hardly pass a 420- μm sieve.

Weigh about 50 g of Weakly Basic Anion - exchange Resin, immerse in water, allow to stand for 30 minutes, and pour the resin with water into a glass tube for chromatography (about 2.5 cm in internal diameter) to prepare the resin column. Pour 500 ml of sodium hydroxide solution (1 25) into the column, and flow at the rate of about 8 ml per minute. Then, pour water to wash the column until the washings show neutrality with phenolphthalein TS, and perform the following test:

Measure 10 ml of the resin, pour it with water into a glass tube for chromatography (1.5 cm in internal diameter), and flow 70 ml of 0.1 mol/l hydrochloric acid from the column at the rate of about 2 ml per minute. The pH of the outflow is 4.0 - 8.0.

***p*-Anisaldehyde** See 4-Methoxy Benzaldehyde

***p*-Anisaldehyde - Sulfuric Acid TS** See 4-Methoxy-benzaldehyde - Sulfuric Acid TS

***p*-Anisidine** $\text{C}_7\text{H}_9\text{NO}$ *p*-Anisidine occurs as white to light brownish crystals or crystalline powder. It is slightly soluble in water, and freely soluble in methanol

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and ethanol.

Melting point: 57 - 60

Note: *p*-anisidine is toxic if inhaled or absorbed through the skin and should be used with due caution.

***p*-Anisidine -Phthalic acid TS** Weigh 1.23 g of *p*-anisidine and 1.66 g of phthalic acid, and dissolve in methanol to make 100 ml. Stopper tightly the solution in light-resistant container, and store in cold place.

Anthrone C₁₄H₁₀O (Guaranteed)

Anthrone TS Weigh 0.05 - 0.2 g of anthrone, and dissolve in 100 ml of diluted sulfuric acid (1 : 20). Prepare freshly before use.

Antimony Trichloride SbCl₃ (Guaranteed)

Antimony Trichloride TS Wash the surface of antimony trichloride with dehydrated chloroform until the washings become clear. Add dehydrated chloroform to antimony trichloride to make a saturated solution.

Store in a light-resistant, tight container, in a cold place. Prepare freshly before use.

Aqua Regia Mix 3 parts hydrochloric acid and 1 part nitric acid by volume. Prepare freshly before use.

Aqueous Ammonia NH₄OH (Guaranteed, Specific gravity: about 0.90)

L-Arabinitol C₅H₁₂O₅ L-Arabinitol occurs as white crystals or crystalline powder.

Clarity of solution: Clear (1.0 g, water 20 ml)

Melting Point: 102 - 104 °C

Water: Not more than 0.5 % (1.0 g, direct titration).

Residue on Ignition: Not more than 0.10 % (2 g).

L-Arginine Hydrochloride H₂N(HN)CNH(CH₂)₃CH(NH₂)COOH · HCl

(L-Arginine Monohydrochloride, Guaranteed)

Arsenic Trioxide See Diarsenic Trioxide.

Arsenic Trioxide (Standard reagent) See Diarsenic Trioxide (Standard reagent).

Arsenic Trioxide TS Weigh 1 g of diarsenic trioxide, and dissolve in 30 ml of sodium hydroxide solution (1 : 40) while heating. Cool, and add slowly acetic acid to make 100 ml.

Arsenic - free Hydrochloric Acid See Hydrochloric Acid, Arsenic - free.

Arsenic - free Zinc See Zinc, Arsenic- free.

L-Ascorbic Acid C₆H₈O₆ "L-Ascorbic Acid"

Ascorbic Acid for Iron Limit Test C₆H₈O₆ (L-Ascorbic Acid, Guaranteed)

Aspartame C₁₄H₁₈N₂O₅ "Aspartame"

L-α-Aspartyl-D-phenylalanine Methyl Ester C₁₄H₁₈N₂O₅

L-α-Aspartyl-D-phenylalanine Methyl Ester occurs as a white crystalline powder. It is soluble in water.

Melting point: 142.0 - 145.0

Purity: Amino acids and peptide compounds Use

L-α-Aspartyl-D-phenylalanine Methyl Ester solution (1 : 1,000) as the sample solution and a mixture of chloroform - methanol - water - acetic acid (32 : 15 : 3 : 1) as the developing solvent. Perform Thin-Layer Chromatography with 2 μl of the sample solution without using a control solution. Only one spot is observed. For the thin - layer plate, use silica gel

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for thin - layer chromatography dried at 110 for 1 hour. When the solvent front rises about 10 cm high, stop the development, air-dry, and dry at 80 for 30 minutes. Spray with ninhydrin TS, dry at 80 for 10 minutes, and observe under daylight.