

20. Melting Point Determination

The melting point is defined to be the temperature at which a crystalline substance melts during heating, when the solid phase and the liquid phase are in an equilibrium. However, it is conventionally defined to be the temperature at which the remaining solid sample melts completely when it is subjected to continuous heating and the change of the sample state that accompanies heating is accurately observed. Since a pure substance has an intrinsic melting point, it is used for the identification and/or confirmation of a substance and also as an indicator of the purity of a substance.

The melting point is the temperature measured by either of the following methods. Method 1 is applied to those substances of which the purity is comparably high and which can be pulverized, Method 2 to those substances which are insoluble in water and can not be readily pulverized, and Method 3 can be applied to polymer resins or synthetic fibers.

Method 1

This method is applied to those substances of which the purity is comparably high and which can be pulverized.

Apparatus

Use as shown in the figure. Alternatively, apparatus in which some of the procedures, such as stirring, heating, and cooling are automated, can be used.

Bath fluid: Usually use clear silicone oil having a viscosity of 50 to 100 mm²/s at an ordinary temperature.

Thermometer with an immersion line: There are six types of thermometers, Type 1- Type 6, which are specified by an appropriate measuring temperature range. For melting points lower than 50°C, use a thermometer Type 1; for 40°C to 100°C, Type 2; for 90°C to 150°C, Type 3; for 140°C to 200°C, Type 4; for 190°C to 250°C, Type 5; for 240°C to 320°C, Type 6.

Capillary tube: Use a hard glass capillary tube 120 mm long, 0.8 to 1.2 mm in inner diameter and 0.2 to 0.3 mm thick, with one end closed.

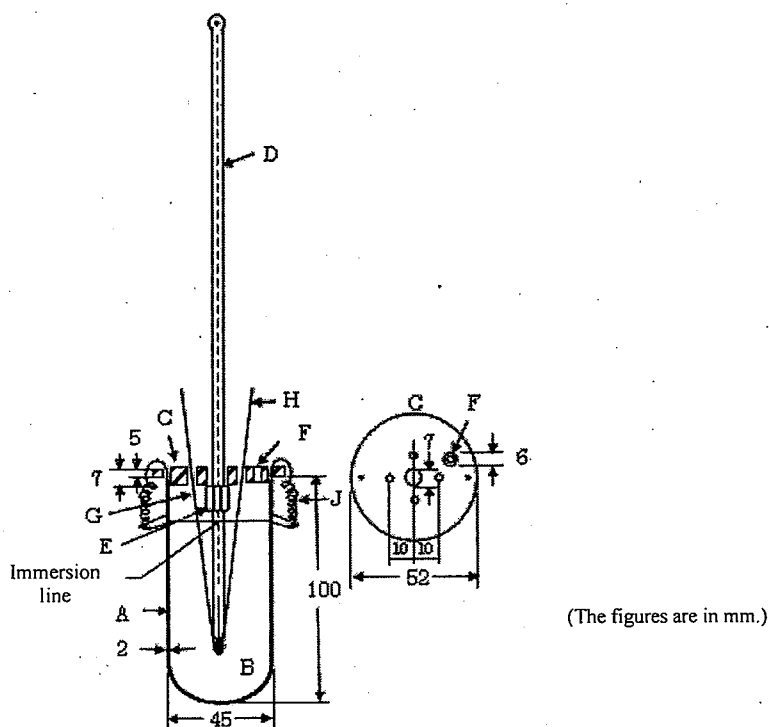


Figure Melting point determination device

- A: Heating vessel of hard glass
- B: Bath fluid
- C: Teflon stopper
- D: Thermometer with an immersion line
- E: Thermometer-fastening spring
- F: Vent for adjustment of the bath fluid volume
- G: Coil spring
- H: Capillary tube
- J: Spring for fastening Teflon stopper

Procedure

Pulverize the sample to a fine powder, and, unless otherwise specified, dry in a desiccator (silica gel) for 24 hours. When it is specified to do the test after drying, dry the sample under the conditions specified in the monograph before measurement.

Place the sample in a dried capillary tube H, and pack it tightly so as to form a layer about 2.5-3.5 mm high by dropping the capillary repeatedly, with the closed end of H down, through a glass tube, about 70 cm long, held vertically on a glass or porous plate.

Heat the bath fluid B until the temperature rises to about 10°C below the expected melting point, place the thermometer D in the bath with the immersion line at the same level as the meniscus of the bath fluid, and insert capillary tube H into a coil spring G so that the packed sample is placed in a position corresponding to the center of the mercury bulb of the thermometer D. Continue heating to raise the temperature at a rate of

approximately 3°C per minute until the temperature rises to 5°C below the expected melting point, then carefully regulate the rate of temperature increase to 1°C per minute.

Read the thermometer indication of the instantaneous temperature at which the sample liquefies completely and no solid is detectable in the capillary, and designate the indicated temperature as the melting point of the sample specimen.

System suitability test

Confirmation of the system suitability of the apparatus should be done periodically by using the Melting Point Standards. The Reference Standard is prepared for the suitability test of the apparatus when it is used with Type 2 - Type 5 thermometers, and consists of 6 highly purified substances: acetanilide, acetophenetidine, caffeine, sulfanilamide, sulfapyridine, and vanillin. The label shows the certified melting points of the respective substances (the end point of the melting change), MP_f . After selecting one of the thermometers and the appropriate Melting Point Standard based upon the expected melting point of a sample specimen, perform a melting point measurement of the selected Reference Standard, according to the above procedure. When the value of the obtained melting point of the Reference Standard is within $MP_f \pm 0.5^\circ\text{C}$ in the case of vanillin and acetanilide, within $MP_f \pm 0.8^\circ\text{C}$ in the case of acetophenetidine and sulfanilamide, and within $MP_f \pm 1.0^\circ\text{C}$ in the case of sulfapyridine and caffeine, the apparatus is assumed to be suitable. The above-mentioned measurement is repeated 3 times and the average is determined to be the melting point of the Reference Standard tested.

When the above suitability test criteria are not met in a certain melting point measurement system of an apparatus and a Reference Standard, do the test again, after checking the packing of the sample specimen into the capillary tube, the locations and positioning of the thermometer and the capillary tube, the heating and stirring of the bath fluid, and the control of the temperature increasing rate. When a melting point measurement system does not meet the suitability test criteria again after checking these measuring conditions, the thermometer with an immersion line should be calibrated again or replaced with a new one.

Method 2

This method is applied to substances such as fats, fatty acids, paraffins or waxes.

Apparatus

Instead of the apparatus specified in Method 1, use a water-containing beaker as a bath fluid and a heating vessel. In this measurement, total immersion mercury-filled thermometers can also be used in place of the thermometer with an immersion line. Furthermore, the capillary tube should be the same as specified in Method 1, except that both ends of the tube are open.

Procedure

Carefully melt the sample at as low a temperature as possible, and, taking care to prevent bubbles, introduce it into a capillary tube to a height of about 10 mm. Allow the capillary containing the sample to stand for 24 hours at below 10°C, or for at least 1 hour in contact with ice, holding the capillary so that the sample can not flow out. Then attach the capillary to the thermometer by means of a rubber band so that the absorbed sample is located at a position corresponding to the center of the mercury bulb. Adjust the capillary tube in a water-containing beaker to such a position that the lower edge of the sample is located 30 mm below the water surface. Heat the beaker with constant stirring until the temperature rises to 5°C below the expected melting point. Then regulate the rate of temperature increase to 1°C per minute. The temperature at which the sample begins floating in the capillary is taken as the melting point of the sample specimen.

Method 3

Melting point can be determined by "Thermal Analysis", either "Differential Thermal Analysis (DTA)" or "Differential Scanning Calorimetry (DSC)".

Thermal Analysis is a generic term for a variety of techniques to measure the physical properties of a substance as a function of temperature and/or time.

Among the physical properties, phase transitions such as solid/liquid phase transition (melting, freezing) and crystal polymorphism or thermal behavior such as heat evolution or absorption accompanying thermal degradation or chemical reaction can be detected by the techniques of differential thermal analysis (DTA) or differential scanning calorimetry (DSC).

DTA is a method for detecting the thermal behavior of a specimen in terms of the temperature change, while DSC employs the heat quantity (enthalpy) change.

Apparatus

Apparatus for DTA or DSC is usually composed of a heating furnace, a temperature-controller, a detector, a device for controlling the atmosphere, and an indicator/recorder.

In a DTA apparatus, a sample specimen and an inert reference material placed in the heating furnace are heated or cooled at a constant rate, and the temperature difference evolved between the sample and reference material is detected continuously by a device such as a thermocouple and recorded as a function of time and/or temperature. As an inert reference material, α -Alumina for thermal analysis is usually adopted.

Two kinds of DSC apparatus, based upon different principles are available as shown below.

(1) Input compensation-type differential scanning calorimetry (Input compensation DSC)

A sample specimen and the reference material in twin furnaces are programmed to be heated or cooled at a constant rate, and the temperature difference between the sample and the reference, which is detected by a device such as a platinum resistance thermometer, is kept at null by controlling the heating unit with a compensation feed-back circuit. The instrument is designed to measure and record continuously the balance of thermal energy applied to each furnace as a function of temperature and/or time.

(2) Heat flux-type differential scanning calorimetry (Heat flux DSC)

A sample specimen and the reference material in twin furnaces are programmed to be heated or cooled at a constant rate, and the temperature difference between the sample and the reference is detected as a difference of heat flux and recorded as a function of temperature and/or time. In heat flux DSC, thermal conductors are adopted so that the heat flux between the sample and the heat reservoir is proportional to the temperature difference between them.

In usual DSC analysis, α -Alumina is used as a reference material, both in Input compensation DSC and in Heat flux DSC. But in some cases, an empty sample container can also be used without any reference material.

Procedure

A sample specimen and the reference material are put in sample pans, and the furnace is heated or cooled under a controlled temperature program. As the temperature changes, the temperature difference (DTA) or heat quantity change (DSC) that develops between the specimen and the reference is detected and recorded

continuously. Apparatus equipped with a data-processor is operated according to the instruction manual provided with the instrument.

A preliminary experiment is needed to determine the appropriate temperature range of measurement, within which a predicted physical change such as melting or polymorphic phase transition will occur, and to confirm that unpredicted thermal changes are not induced in a specimen in that temperature range. In this preliminary test, a wide temperature range (room temperature-the temperature at which degradation begins) can be scanned at a rapid heating rate (10-20°C/min). Thereafter, tests by DSC or DTA should be performed at a low heating rate, usually 2°C/min, in the chosen temperature range. However, when a clear heat change cannot be observed, such as in a case of glass-transition, the heating rate may be changed to a higher or a lower rate, as appropriate for the kind of physical change being observed. By analyzing the measured DTA-curve or DSC-curve, a quantity of heat change and/or a specific temperature (ignition, peak or end temperature) that accompanies a physical change, such as melting or polymorphic phase transition, can be obtained.

Calibration of apparatus

(1) Temperature calibration

Temperature calibration for DTA and/or DSC apparatus can be performed by using reference substances having an intrinsic thermal property, such as melting point of pure metals or organic substances, or phase transition point of crystalline inorganic salts or oxides. Melting points of Indium for thermal analysis and/or Tin for thermal analysis are usually employed for calibration.

(2) Heat-quantity calibration for DSC

For accurate estimation of a quantity of heat change (enthalpic change) of a sample specimen, caused by a certain physical change accompanying a temperature change, it is necessary to calibrate the apparatus by using appropriate reference substances. As indicated in the section of Temperature calibration, heat-quantity calibration for DSC apparatus can be performed by using appropriate reference substances having a known definite enthalpic change caused by such physical changes as melting of pure metals and/or organic substances, or phase transition of crystalline inorganic salts. Melting points of Indium for thermal analysis and/or Tin for thermal analysis are usually employed for calibration.

Notes on operating conditions

When DTA or DSC measurements are made, the following items must be recorded: sample size, discrimination of open or closed-type sample container, heating or cooling rate, measuring temperature range, and kind and flow rate of atmospheric gas.

21. Readily Carbonizable Substances Test

Readily Carbonizable Substances Test is a method to examine the minute impurities contained in samples, which are readily colored by addition of sulfuric acid.

Procedure

Use 94.5% to 95.5% sulfuric acid (sulfuric acid for readily carbonizable substances). Before use, wash the Nessler tubes thoroughly with sulfuric acid. Unless otherwise specified, proceed as follows. When the sample

is solid, place 5 mL of sulfuric acid in a Nessler tube, to which add a quantity of the finely powdered sample, little by little, as directed in the monograph, and dissolve it completely by stirring with a glass rod. When the sample is liquid, transfer a volume of the sample, as directed in the monograph, to a Nessler tube, add 5 mL of sulfuric acid, and mix by shaking. If the temperature of the content of the tube rises, cool the content; maintain it at the standard temperature, if the reaction may be affected by the temperature. Allow to stand for 15 minutes, and compare the color of the liquid with that of the matching fluid in the Nessler tube specified in the monograph, by viewing transversely against a white background.

When it is specified to heat the sample with sulfuric acid, transfer the sample and sulfuric acid to a Nessler tube, heat as specified, and compare the colors.

22. Standard Solutions, Matching Fluids for Color, Reference Standards, Reagents, Test Solutions, Measuring Instruments and Appliances

Standard Solutions for Volumetric Analysis are the solutions of reagent with an accurately known concentration, mainly used for the volumetric analysis.

Standard Solutions are used as the standard for the comparison in the tests specified in the JSNM.

Matching Fluids for Color are used as the reference for the comparison of color in the tests specified in the JSNM.

Reference Standards are the substances prepared to a specified purity or quality necessary with regard to their intended use as prescribed in monographs of the JSNM.

Reagents are the substances used for the tests specified in the JSNM. The reagents that are described as “standard reagent”, “special class”, “first class”, “for pH determination”, etc. in the JSNM meet the corresponding specifications under the Japan Industrial Standards (JIS). The tests for them shall be performed according to the test methods under the JIS. In the case where the reagent names in the JSNM differ from those of the JIS, the JIS names are given in the brackets. The reagents that are described as “JP reference standard” or “JP monograph” meet the specifications of the corresponding reference standards or monographs. In the case of the reagents that are described merely as test items, the corresponding test methods of the JP are to be applied.

Test Solutions are the solutions prepared for use in the tests of the JSNM.

Measuring Instruments are the instruments or machines used for measuring mass or volume in the tests of the JSNM.

Appliances are the instruments specified in order to make test conditions as consistent as possible in the tests of the JSNM.

(1) Volumetric Standard Solutions (VS)

Hydrochloric Acid, 0.5 mol/L

1000 mL of this solution contains 18.230 g of hydrochloric acid (HCl: 36.46).

Preparation: Dilute 45 mL of hydrochloric acid with water to make 1000 mL, and standardize the solution as follows:

Standardization: Weigh accurately about 0.5 g of sodium carbonate (standard reagent), previously heated between 500°C and 650°C for 40 to 60 minutes and allowed to cool in a desiccator (silica gel). Dissolve it in 50 mL of water, and titrate with the prepared hydrochloric acid to calculate the molarity factor (indicator: 3 drops of methyl red TS). In the indicator method, when the end point is approached, boil the solution carefully, stopper the flask loosely, allow to cool, and continue the titration until the color of the solution changes to persistent orange to orange-red.

Each mL of 0.5 mol/L hydrochloric acid VS = 26.50 mg of Na₂CO₃

Hydrochloric Acid, 0.2 mol/L

1000 mL of this solution contains 7.292 g of hydrochloric acid (HCl: 36.46).

Preparation: Dilute 18 mL of hydrochloric acid with water to make 1000 mL, and standardize the solution as follows:

Standardization: Weigh accurately about 0.15 g of sodium carbonate (standard reagent), previously heated between 500°C and 650°C for 40 to 60 minutes and allowed to cool in a desiccator (silica gel). Dissolve it in 30 mL of water, and titrate the solution with the prepared hydrochloric acid to calculate the molarity factor (indicator: 3 drops of methyl red TS). In the indicator method, when the end point is approached, boil the solution carefully, stopper the flask loosely, allow to cool, and continue the titration until the color of the solution changes to persistent orange to orange-red.

Each mL of 0.2 mol/L hydrochloric acid VS = 10.60 mg of Na_2CO_3

Hydrochloric Acid, 0.1 mol/L

1000 mL of this solution contains 3.6461 g of hydrochloric acid (HCl: 36.46).

Preparation: Before use, dilute 0.2 mol/L hydrochloric acid VS with water to make exactly 2 times the initial volume.

Each mL of 0.1 mol/L hydrochloric acid VS = 5.300 mg of Na_2CO_3

Hydrochloric Acid, 0.01 mol/L

1000 mL of this solution contains 0.36461 g of hydrochloric acid (HCl: 36.46).

Preparation: Before use, dilute 0.2 mol/L hydrochloric acid VS with water to make exactly 20 times the initial volume.

Potassium Permanganate, 0.02 mol/L

1000 mL of this solution contains 3.1607 g of potassium permanganate (KMnO_4 : 158.03).

Preparation: Dissolve 3.2 g of potassium permanganate in water to make 1000 mL, and boil the solution for 15 minutes. Allow the solution to stand for at least 48 hours in a tightly stoppered flask, and filter it through a glass filter (G3 or G4). Standardize the solution as follows:

Standardization: Weigh accurately about 0.3 g of sodium oxalate (standard reagent), previously dried between 150°C and 200°C for 1 to 1.5 hours and allowed to cool in a desiccator (silica gel), transfer it to a 500 mL conical flask, dissolve in 30 mL of water, add 250 mL of diluted sulfuric acid (1 in 20), and warm the mixture between 30°C and 35°C. Transfer the prepared potassium permanganate solution to a burette, add quickly 40 mL of the solution under gentle stirring from the burette, and allow to stand until the red color of the mixture disappears. Warm the solution between 55°C and 60°C, and complete the titration with the potassium permanganate solution until a faint red color persists for 30 seconds. Add the last 0.5 to 1 mL dropwise before the end point, being particularly careful to allow the solution to be decolorized before the next drop is added. Calculate the molarity factor.

Each mL of 0.02 mol/L potassium permanganate VS = 6.700 mg of $\text{Na}_2\text{C}_2\text{O}_4$

Note: Store protected from light. This solution, if stored for a long period, should be restandardized.

Potassium Permanganate, 0.002 mol/L

1000 mL of this solution contains 0.31607 g of potassium permanganate (KMnO_4 : 158.03).

Preparation: Before use, dilute 0.02 mol/L potassium permanganate VS with water to make exactly 10 times the initial volume.

Potassium Hydroxide, 0.1 mol/L

1000 mL of this solution contains 5.611 g of potassium hydroxide (KOH: 56.11).

Preparation: Dissolve 6.5 g of potassium hydroxide in 950 mL of water. Add a freshly prepared, saturated solution of barium hydroxide octahydrate until no more precipitate is produced. Shake the mixture thoroughly, and allow it to stand for 24 hours in a tightly stoppered bottle. Decant the supernatant liquid or filter the solution through a glass filter (G3 or G4), and standardize the solution as follows:

Standardization: Weigh accurately about 0.25 g of amidosulfuric acid (sulfamic acid)(standard reagent), previously dried in a desiccator (in vacuum, silica gel) for 24 to 48 hours. Dissolve it in 25 mL of freshly boiled and cooled water, and add 2 drops of bromothymol blue TS. Titrate with the prepared potassium hydroxide solution until it acquires a green color. Calculate the molarity factor.

Each mL of 0.1 mol/L potassium hydroxide VS =9.709 mg of HOSO_2NH_2

Note: Store in tightly stoppered bottles or in containers provided with a carbon dioxide-absorbing tube (soda lime). This solution, if stored for a long period, should be restandardized.

Potassium Hydroxide-Ethanol, 0.5 mol/L

1000 mL of this solution contains 28.055 g of potassium hydroxide (KOH: 56.11).

Preparation: Dissolve 35 g of potassium hydroxide in 20 mL of water, and add aldehyde-free ethanol to make 1000 mL. Allow the solution to stand for 24 hours in a tightly stoppered bottle. Then quickly decant the supernatant liquid, and standardize the solution as follows:

Standardization: Measure exactly 25 mL of 0.25 mol/L sulfuric acid VS, add 50 mL of water, and titrate with the prepared potassium hydroxide-ethanol solution to calculate the molarity factor (indicator: 2 drops of phenolphthalein TS). In the indicator method, titrate until the solution acquires a light red color.

Note: Store in tightly stoppered bottles, protected from light. Standardize before use.

Potassium Hydroxide-Ethanol, 0.1 mol/L

1000 mL of this solution contains 5.611 g of potassium hydroxide (KOH: 56.11).

Preparation: Dissolve 7 g of potassium hydroxide in 20 mL of water, and add aldehyde-free ethanol to make 1000 mL. Allow the solution to stand for 24 hours in a tightly stoppered bottle. Then quickly decant the supernatant liquid, and standardize the solution as follows:

Standardization: Measure exactly 15 mL of 0.05 mol/L sulfuric acid VS, add 50 mL of water, and titrate with the prepared potassium hydroxide-ethanol solution to calculate the molarity factor (indicator: 2 drops of phenolphthalein TS). In the indicator method, titrate until the solution acquires a light red color.

Note: Store in tightly stoppered bottles, protected from light. Standardize before use.

Sodium Hydroxide, 1 mol/L

1000 mL of this solution contains 39.997 g of sodium hydroxide (NaOH: 40.00).

Preparation: Dissolve 42 g of sodium hydroxide in 950 mL of water. Add a freshly prepared, saturated solution of barium hydroxide octahydrate until no more precipitate is produced. Mix well the mixture, and allow to stand for 24 hours in a tightly stoppered bottle. Decant the supernatant liquid or filter the solution through a glass filter (G3 or G4), and standardize the solution as follows:

Standardization: Weigh accurately about 1.5 g of amidosulfuric acid (sulfamic acid)(standard reagent), previously dried in a desiccator (in vacuum, silica gel) for 24 to 48 hours. Dissolve it in 25 mL of freshly

boiled and cooled water, and titrate the solution with the prepared sodium hydroxide solution to calculate the molarity factor (indicator method: 2 drops of bromothymol blue TS). In the indicator method, titrate until the solution acquires a green color.

Each mL of 1 mol/L sodium hydroxide VS = 97.09 mg of HOSO_2NH_2

Note: Store in tightly stoppered bottles or in containers provided with a carbon dioxide-absorbing tube (soda lime). This solution, if stored for a long period, should be restandardized.

Sodium Hydroxide, 0.5 mol/L

1000 mL of this solution contains 19.999 g of sodium hydroxide (NaOH: 40.00).

Preparation: Dissolve 22 g of sodium hydroxide in 950 mL of water. Add a freshly prepared, saturated solution of barium hydroxide octahydrate until no more precipitate is produced. Mix well the mixture, and allow to stand for 24 hours in a tightly stoppered bottle. Decant the supernatant liquid or filter the solution through a glass filter (G3 or G4), and standardize the solution as follows:

Standardization: Weigh accurately about 0.7 g of amidosulfuric acid (sulfamic acid) (standard reagent), previously dried in a desiccator (in vacuum, silica gel) for 24 to 48 hours. Dissolve it in 25 mL of freshly boiled and cooled water, and titrate the solution with the prepared sodium hydroxide solution to calculate the molarity factor (indicator method: 2 drops of bromothymol blue TS). In the indicator method, titrate until the solution acquires a green color.

Each mL of 0.5 mol/L sodium hydroxide VS = 48.55 mg of HOSO_2NH_2

Note: Store in tightly stoppered bottles or in containers provided with a carbon dioxide-absorbing tube (soda lime). This solution, if stored for a long period, should be restandardized.

Sodium Hydroxide, 0.1 mol/L

1000 mL of this solution contains 3.9997 g of sodium hydroxide (NaOH: 40.00).

Preparation: Dissolve 4.5 g of sodium hydroxide in 950 mL of water. Add a freshly prepared, saturated solution of barium hydroxide octahydrate until no more precipitate is produced. Mix well the mixture, and allow to stand for 24 hours in a tightly stoppered bottle. Decant the supernatant liquid or filter the solution through a glass filter (G3 or G4), and standardize the solution as follows:

Standardization: Weigh accurately about 0.15 g of amidosulfuric acid (sulfamic acid) (standard reagent), previously dried in a desiccator (in vacuum, silica gel) for 24 to 48 hours. Dissolve it in 25 mL of freshly boiled and cooled water, and titrate the solution with the prepared sodium hydroxide solution to calculate the molarity factor (indicator method: 2 drops of bromothymol blue TS). In the indicator method, titrate until the solution acquires a green color.

Each mL of 0.1 mol/L sodium hydroxide VS = 9.709 mg of HOSO_2NH_2

Note: Store in tightly stoppered bottles or in containers provided with a carbon dioxide-absorbing tube (soda lime). This solution, if stored for a long period, should be restandardized.

Sodium Thiosulfate, 0.2 mol/L

1000 mL of this solution contains 49.636 g of sodium thiosulfate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$: 248.18).

Preparation: Dissolve 52 g of sodium thiosulfate and 0.2 g of anhydrous sodium carbonate in freshly boiled and cooled water to make 1000 mL, and standardize the solution as follows:

Standardization: Weigh accurately about 0.2 mg of potassium iodate (standard reagent), previously dried

between 120°C and 140°C for 2 hours and allowed to cool in a desiccator (silica gel), and transfer to an iodine flask. Dissolve it in 25 mL of water, add 4 g of potassium iodide and 10 mL of dilute sulfuric acid, and stopper the flask. After allowing the mixture to stand for 10 minutes, add 100 mL of water, and titrate the liberated iodine with the prepared sodium thiosulfate solution to calculate the molarity factor. When the solution assumes a pale yellow color as the end point is approached, add 3 mL of starch TS. Continue the titration until the blue color disappears. Perform a blank determination in the same manner, and make any necessary correction.

Each mL of 0.2 mol/L sodium thiosulfate VS = 7.133 mg of KIO_3

Note: This solution, if stored for a long period, should be restandardized.

Sodium Thiosulfate, 0.1 mol/L

1000 mL of this solution contains 24.818 g of sodium thiosulfate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$: 248.18).

Preparation: Before use, dilute 0.2 mol/L sodium thiosulfate VS with freshly boiled and cooled water to make 2 times the initial volume.

Sodium Thiosulfate, 0.01 mol/L

1000 mL of this solution contains 2.4818 g of sodium thiosulfate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$: 248.18).

Preparation: Before use, dilute 0.2 mol/L sodium thiosulfate VS with freshly boiled and cooled water to make 20 times the initial volume.

Sulfuric Acid, 0.25 mol/L

1000 mL of this solution contains 24.520 g of sulfuric acid (H_2SO_4 : 98.08).

Preparation: Add slowly, under stirring, 15 mL of sulfuric acid to 1000 mL of water, allow to cool, and standardize the solution as follows:

Standardization: Weigh accurately about 0.4 g of sodium carbonate (standard reagent), previously heated between 500°C and 650°C for 40 to 60 minutes and allowed to cool in a desiccator (silica gel). Dissolve it in 50 mL of water, and titrate with the prepared sulfuric acid to calculate the molarity factor (indicator: 3 drops of methyl red TS). In the indicator method, when the end point is approached, boil the solution carefully, stopper the flask loosely, allow to cool, and continue the titration until the color of the solution changes to persistent orange to orange-red.

Each mL of 0.25 mol/L sulfuric acid VS = 26.50 mg of Na_2CO_3

Sulfuric Acid, 0.05 mol/L

1000 mL of this solution contains 4.904 g of sulfuric acid (H_2SO_4 : 98.08).

Preparation: Add slowly, under stirring, 3 mL of sulfuric acid to 1000 mL of water, allow to cool, and standardize the solution as follows:

Standardization: Weigh accurately about 80 mg of sodium carbonate (standard reagent), previously heated between 500°C and 650°C for 40 to 60 minutes and allowed to cool in a desiccator (silica gel). Dissolve it in 30 mL of water, and titrate the solution with the prepared sulfuric acid to calculate the molarity factor (indicator: 3 drops of methyl red TS). In the indicator method, when the end point is approached, boil the solution carefully, stopper the flask loosely, allow to cool, and continue the titration until the color of the solution changes to persistent orange to orange-red.

Each mL of 0.05 mol/L sulfuric acid VS= 5.300 mg of Na_2CO_3

(2) Standard Solutions

Borate pH Standard Solution See pH Determination.

Calcium Hydroxide pH Standard Solution See pH Determination.

Carbonate pH Standard Solution See pH Determination.

Oxalate pH Standard Solution See pH Determination.

Phosphate pH Standard Solution See pH Determination.

Phthalate pH Standard Solution See pH Determination.

Standard Arsenic Solution Pipet 10 mL of Standard Arsenic Stock Solution, add 10 mL of dilute sulfuric acid, and add freshly boiled and cooled water to make exactly 1000 mL. Each mL of the solution contains 1 μg of arsenic (III) trioxide (As_2O_3). Prepare before use and preserve in a glass-stoppered bottle.

Standard Arsenic Stock Solution Weigh accurately 0.100 g of finely powdered arsenic (III) trioxide dried at 105°C for 4 hours, and add 5 mL of sodium hydroxide solution (1 in 5) to dissolve. Add dilute sulfuric acid to neutralize, add further 10 mL of dilute sulfuric acid, and add freshly boiled and cooled water to make exactly 1000 mL.

Standard Iron Solution Weigh exactly 86.3 mg of ammonium iron (III) sulfate dodecahydrate, dissolve in 100 mL of water, and add 5 mL of dilute hydrochloric acid and water to make exactly 1000 mL. Each mL of this solution contains 0.01 mg of iron (Fe).

Standard Lead Solution Measure exactly 10 mL of Standard Lead Stock Solution, and add water to make exactly 100 mL. Prepare before use. Each mL of this solution contains 0.01 mg of lead (Pb).

Standard Lead Stock Solution Weigh exactly 159.8 mg of lead (II) nitrate, dissolve in 10 mL of dilute nitric acid, and add water to make exactly 1000 mL. Prepare and store this solution using glass containers, free from soluble lead salts.

Standard Liquids for Calibrating Viscosimeters [JIS, Standard Liquids for Calibrating Viscosimeters (Z8809)]

Standard Lithium Solution for Atomic Absorption Spectrophotometry Weigh exactly 6.107 g of lithium chloride (anhydrous) and dissolve in 0.01 mol/L hydrochloric acid TS to make exactly 1000 mL. Each mL of this solution contains 1.00 mg of lithium (Li).

Standard Vinyl Chloride Solution Transfer ethanol to a 200-mL volumetric flask in a volume of 2 to 3 mL smaller than the marked volume, stopper with a silicone rubber stopper and measure the mass. Inject about 200 mg of liquefied vinyl chloride through the silicone rubber stopper to dissolve in ethanol. Then, measure the weight, determine the increase in mass exactly, and define this value as "a." Add ethanol to make exactly 200 mL through the silicone rubber stopper, shake well to make uniform, and use this solution as the standard vinyl chloride stock solution. Cool the standard vinyl chloride stock solution in a methanol-dry ice bath, transfer 95 mL of cooled ethanol in a 100-mL volumetric flask in the same manner, add 1 mL of the standard stock solution while cooling, and allow to stand to room temperature. Then, add ethanol to make 100 mL, and use this solution as the vinyl chloride standard solution. The concentration of the standard stock solution is

a/(100 × 1.0567) ppm. Dilute the standard solution with ethanol to prepare standard solutions for calibration curve with a concentration of 0.1 to 10 ppm.

(3) Matching Fluids for Color

Cobaltous Chloride Colorimetric Stock Solution

Weigh 65 g of cobaltous chloride (cobalt (II) chloride hexahydrate), and dissolve in 25 mL of hydrochloric acid and water to make 1000 mL. Pipet 5 mL of this solution into a 250-mL iodine flask. Add 5 mL of hydrogen peroxide TS and 15 mL of a solution of sodium hydroxide (1 in 5), and boil for 10 minutes. Cool, and add 2 g of potassium iodide and 20 mL of diluted sulfuric acid (1 in 4). When the precipitate dissolves, titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS).

Each mL of 0.1 mol/L sodium thiosulfate VS = 23.793 mg of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$

According to the titration value, add diluted hydrochloric acid (1 in 40) to make a solution containing 59.5 mg of cobaltous chloride ($\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$: 237.93) in each mL, and use this solution as the colorimetric stock solution.

Copper Sulfate Colorimetric Stock Solution

Weigh 65 g of copper sulfate (copper (II) sulfate pentahydrate), and dissolve in 25 mL of hydrochloric acid and water to make 1000 mL. Pipet 10 mL of this solution into an iodine flask. Add 4 mL of acetic acid and 3 g of potassium iodide. Titrate the liberated iodine with 0.1 mol/L sodium thiosulfate (indicator: 1 mL of starch TS).

Each mL of 0.1 mol/L sodium thiosulfate VS = 24.968 mg of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

According to the titrated value, add diluted hydrochloric acid (1 in 40) to make a solution containing 62.4 mg of copper sulfate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$: 249.68) in each mL, and use this solution as the colorimetric stock solution.

Ferric Chloride Colorimetric Stock Solution

Weigh 55 g of ferric chloride (iron (III) chloride hexahydrate), and dissolve in 25 mL of hydrochloric acid and add water to make 1000 mL. Measure exactly 10 mL of this solution, transfer to an iodine flask, add 15 mL of water and 3 g of potassium iodide, stopper tightly, and allow to stand in a dark place for 15 minutes. Add 100 mL of water to the mixture, and titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 1 mL of starch TS).

Each mL of 0.1 mol/L sodium thiosulfate VS = 27.03 mg of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$

According to the titrated value, add diluted hydrochloric acid (1 in 40) to make a solution containing 45.0 mg of ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$: 270.30) in each mL, and use this solution as the colorimetric stock solution.

(4) Reference Standards, Reagents and Test Solutions (TS)

Acetanilide reference standard for melting point determination [JP Acetanilide reference standard for melting point determination]

Acetic acid 2-methoxyethyl $\text{CH}_3\text{COOCH}_2\text{CH}_2\text{OCH}_3$

Melting point: -65.1°C , Boiling point: 145°C

Acetic acid, dilute Dilute 6 g of acetic acid (100) with water to make 100 mL (1 mol/L).

Acetic acid, glacial See acetic acid (100).

Acetic acid (100) CH_3COOH [K8355, Acetic acid, Special class]

Acetic anhydride $(\text{CH}_3\text{CO})_2\text{O}$ [K8886, Special class]

Acetone CH_3COCH_3 [K8034, Special class]

Acetonitrile CH_3CN [K8032, Special class]

Acetonitrile for liquid chromatography CH_3CN Colorless clear liquid. Mixable with water.

Purity Ultraviolet light absorbing substances: Determine the absorbances at the following wavelengths as directed under Ultraviolet-visible Spectrophotometry, using water as the control: not more than 0.07 at 200 nm, not more than 0.046 at 210 nm, not more than 0.027 at 220 nm, not more than 0.014 at 230 nm and not more than 0.009 at 240 nm.

Acetophenetidine reference standard for melting point determination [JP Acetophenetidine reference standard for melting point determination]

Acetylene See dissolved acetylene.

Acidic stannous chloride TS See tin (II) chloride TS, acidic.

Acidic tin (II) chloride TS See tin (II) chloride TS, acidic.

Acrylic acid reference standard Acrylic acid (Special class). It contains not be less than 99.0% (Acrylic acid, $\text{C}_3\text{H}_4\text{O}_2$:72.06).

Assay: Weigh accurately about 1 g of this substance, add 20 mL of water, mix, and titrate with 1 mol/L of sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS).

Each mL of 1 mol/L sodium hydroxide VS = 72.06 mg $\text{C}_3\text{H}_4\text{O}_2$

Aldehyde-free ethanol See ethanol, aldehyde-free.

Alizarin S See alizarin red S.

Alizarin S TS See alizarin red S TS.

Alizarin red S $\text{C}_{14}\text{H}_7\text{NaO}_7\text{S}$ [K8057, Special class] Range of color change: pH (yellow) 3.7-5.2 (orange).

Alizarin red S TS Dissolve 0.1 g of alizarin red S in water to make 100 mL, and filter.

α -Alumina for thermal analysis $\alpha\text{-Al}_2\text{O}_3$ Prepared for thermal analysis.

Amidosulfuric acid (standard reagent) HOSO_2NH_2 [K8005, Amidosulfuric acid, Standard reagent for volumetric analysis] or [K8587, Special class]

Ammonia copper TS To 0.5 g of cupric carbonate monohydrate add 10 mL of water, triturate, and add 10 mL of ammonia solution (28).

Ammonia TS To 400 mL of ammonia solution (28) add water to make 1000 mL (10%).

Ammonium chloride NH_4Cl [K8116, Special class]

Ammonium chloride TS Dissolve 10.5 g of ammonium chloride in water to make 100 mL (2 mol/L).

Ammonium iron (III) sulfate dodecahydrate $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ [K8982, Ammonium iron (III) sulfate dodecahydrate, Special class]

Ammonium peroxodisulfate $(\text{NH}_4)_2\text{S}_2\text{O}_8$ [K8252, Special class]

Ammonium persulfate See ammonium peroxodisulfate.

Ammonium sodium hydrogenphosphate See ammonium sodium hydrogenphosphate tetrahydrate.

Ammonium sodium hydrogenphosphate tetrahydrate $\text{NaNH}_4\text{HPO}_4 \cdot 4\text{H}_2\text{O}$ [K9013, Special class]

Ammonia solution (28) NH_3 [K8085, Ammonia Water, Special class, Specific gravity: about 0.90, Density:

0.908 g/mL, Content: 28–30%]

Ammonium sulfate $(\text{NH}_4)_2\text{SO}_4$ [K8960, Special class]

Ammonium thiocyanate NH_4SCN [K9000, Special class]

Ammonium thiocyanate-cobalt nitrate TS See ammonium thiocyanate-cobalt (II) nitrate TS.

Ammonium thiocyanate-cobalt (II) nitrate TS Dissolve 17.4 g of ammonium thiocyanate and 2.8 g of cobalt (II) nitrate hexahydrate in water to make 100 mL.

Ammonium thiocyanate TS Dissolve 8 g of ammonium thiocyanate in water to make 100 mL (1 mol/L).

Anhydrous ethanol See ethanol (99.5).

Anhydrous sodium carbonate See sodium carbonate, anhydrous.

Anhydrous sodium sulfate See sodium sulfate, anhydrous.

Arsenic (III) trioxide As_2O_3 [K8044, Arsenic (III) trioxide, Special class]

Arsenic trioxide See arsenic (III) trioxide.

Barium chloride See barium chloride dihydrate.

Barium chloride dehydrate $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ [K8155, Special class]

Barium chloride TS Dissolve 12 g of barium chloride dihydrate in water to make 100 mL (0.5 mol/L).

Barium hydroxide See barium hydroxide octahydrate.

Barium hydroxide octahydrate $\text{Ba}(\text{OH})_2 \cdot 8\text{H}_2\text{O}$ [K8577, Special class] Preserve in tightly stoppered bottles.

Benzene C_6H_6 [K8858, Special class]

Benzyl alcohol $\text{C}_6\text{H}_5\text{CH}_2\text{OH}$ Colorless liquid, having a faint, characteristic odor.

Specific gravity: d_{20}^{20} , 1.042 - 1.053, Purity: not less than 97.0%

Blue litmus paper See litmus paper, blue.

Boric acid H_3BO_3 [K8863, Boric acid, Special class]

Bromothymol blue $\text{C}_{27}\text{H}_{28}\text{Br}_2\text{O}_5\text{S}$ [K8842, Special class] Range of color change: pH (yellow) 6.0-7.6 (blue)

Bromothymol blue TS Dissolve 0.1 g of bromothymol blue in 100 mL of dilute ethanol, and filter if necessary.

Bromoform CHBr_3 (another name: tribromomethane) Colorless liquid. Preserve in a dark, cold place.

Melting point: 7-9°C, boiling point: 149 – 152°C, specific gravity: d^{15} 2.89

1-Butanol $\text{CH}_3(\text{CH}_2)_2\text{CH}_2\text{OH}$ [K8810, Special class]

n-Butanol See 1-butanol.

Caffeine reference standard for melting point determination [JP Caffeine reference standard for melting point determination]

Calcium chloride See calcium chloride dihydrate.

Calcium chloride dihydrate $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ [K8122, Special class]

Calcium chloride TS Dissolve 7.5 g of calcium chloride dihydrate in water to make 100 mL (0.5 mol/L).

Calcium hydroxide $\text{Ca}(\text{OH})_2$ [K8575, Special class]

Calcium hydroxide for pH determination [K8575, Special class] Calcium hydroxide prepared for pH determination. Use the saturated solution obtained at 23-27°C of which pH is 12.45 at 25°C.

Calcium hydroxide TS To 3 g of calcium hydroxide add 1000 mL of cold distilled water, and occasionally shake the mixture vigorously for 1 hour. Allow to stand, and use the supernatant liquid (0.04 mol/L).

Calcium nitrite See calcium nitrate tetrahydrate.

Calcium nitrate tetrahydrate $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ [K8549, Special class]

Carbon tetrachloride CCl_4 [K8459, Special class]

Catechol $\text{C}_6\text{H}_4(\text{OH})_2$ White to gray granules or crystals. It is odorless or has a faint, characteristic odor. Melting point: $103^\circ\text{C} - 107^\circ\text{C}$, Purity: not less than 98.0%.

Chloroform CHCl_3 [K8322, Special class]

Cobalt (II) chloride hexahydrate $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ [K8129, Special class]

Cobalt (II) nitrate hexahydrate $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ [K8552, Special class]

Cobalt chloride See cobalt (II) chloride hexahydrate.

Cobaltous chloride See cobalt (II) chloride hexahydrate.

Cobaltous nitrate See cobalt (II) nitrate hexahydrate.

Copper (II) sulfate pentahydrate $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ [K8983, Special class]

Cupric carbonate See cupric carbonate monohydrate.

Cupric carbonate monohydrate $\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2 \cdot \text{H}_2\text{O}$ A blue to blue-green powder. It is insoluble in water, and dissolves foamingly in dilute acid. It dissolves in ammonia TS and shows a deep blue color.

Purity

(1) Chloride: Not more than 0.036%.

(2) Sulfate: Not more than 0.120%.

(3) Iron: Dissolve 5.0 g of this substance in excess ammonia TS and filter. Wash the residue with ammonia TS, dissolve in dilute hydrochloric acid, add excess ammonia TS and filter. Wash the residue with ammonia TS, and dry to constant mass: the residue is not more than 10 mg.

Cupric sulfate See copper (II) sulfate pentahydrate.

Dibasic sodium phosphate, anhydrous, for pH determination See disodium hydrogen phosphate for pH determination.

Dibenzylamine $(\text{C}_6\text{H}_5\text{CH}_2)_2\text{NH}$ A colorless or yellowish clear liquid, practically insoluble in water, soluble in alcohol and ethanol. Purity: not less than 98.0%.

Specific gravity: 1.027-1.032.

1,2-Dibromoethane $\text{BrH}_2\text{CCH}_2\text{Br}$ Colorless liquid having a characteristic odor.

Melting point: $9^\circ\text{C} - 10^\circ\text{C}$, Boiling point: $131^\circ\text{C} - 132^\circ\text{C}$, d^0 2.21; d^{20} 2.18

1,3-Dibromopropane $\text{Br}(\text{CH}_2)_3\text{Br}$

Diethylene glycol $\text{HO}(\text{CH}_2\text{CH}_2\text{O})_2\text{H}$ Colorless and odorless liquid. Miscible with water and with ethanol (95).

Specific gravity: d_{20}^{20} 1.118-1.120

Diethyl ether $\text{C}_2\text{H}_5\text{OC}_2\text{H}_5$ [K8103, Special class]

Dilute acetic acid See acetic acid, dilute.

Dilute ethanol See ethanol, dilute.

Dilute hydrochloric acid See hydrochloric acid, dilute.

Dilute methylene blue TS See methylene blue TS, dilute.

Dilute nitric acid See nitric acid, dilute.

Dilute sulfuric acid See sulfuric acid, dilute.

N,N-Dimethylacetamide $\text{CH}_3\text{CON}(\text{CH}_3)_2$ A clear, colorless liquid.

Boiling point: 163-165°C, Specific gravity: 0.938-0.945, Water content: not more than 0.2% (0.1 g, Coulometric titration).

Purity: Perform the test with 3 µL of this substance as directed under Gas Chromatography according to the following conditions, and measure the area of each peak using automatic integration. Calculate the amount of N,N-Dimethylacetamide by the area percentage method: not less than 98.0%.

Operating conditions

Detector: Hydrogen flame-ionization detector

Column: A fused silica column, 0.25 mm in the inside diameter and 30 m in length, coated with polyethylene glycol 20M for gas chromatography 0.5 µm in thickness

Column temperature: The sample is injected at a constant temperature of about 70°C, keep this temperature for 1 minute, then raise to 200°C in a rate of 10°C per minute, and keep 200°C for 3 minutes.

Carrier gas: Helium

Flow rate (linear velocity): About 30 cm/sec

Time span of measurement: About 2 times as long as the retention time of N, N-dimethylacetamide.

p-Dimethylaminobenzaldehyde See 4-dimethylaminobenzaldehyde.

4-Dimethylaminobenzaldehyde (CH₃)₂NC₆H₄CHO [K8496, p-Dimethylaminobenzaldehyde, Special class]

N,N-Dimethylformamide HCON(CH₃)₂ [K8500, Special class]

Dimethylformamide See N,N-dimethylformamide.

Dimethylsulfoxide (CH₃)₂SO [K9702, Special class]

Dimethylsulfoxide for ultraviolet-visible spectrophotometry Colorless crystals or clear colorless liquid, having a characteristic odor.

It is highly hygroscopic.

Congealing point: Not less than 18.3°C

Purity: Read absorbance of this substance, immediately after saturating with nitrogen, using water as the blank as directed under Ultraviolet-visible Spectrophotometry: its value is not more than 0.20 at 270 nm, not more than 0.09 at 275 nm, not more than 0.06 at 280 nm, and not more than 0.015 at 300 nm. It exhibits no characteristic absorption between 260 nm and 350 nm.

Water content: Not more than 0.1.

Diphenylmethane diisocyanate C₁₅H₁₀O₂N₂ White to pale yellow solid. Soluble in benzene, toluene, acetone, and ether.

Specific gravity: d_4^{30} 1.19, Boiling point: 190°C (665 Pa)

Disodium hydrogen phosphate dodecahydrate Na₂HPO₄·12H₂O [K9019, Disodium hydrogen phosphate dodecahydrate, Special class]

Disodium hydrogen phosphate for pH determination Na₂HPO₄ [K9020, Disodium hydrogen phosphate, for pH standard solution]

Dissolved acetylene C₂H₂ [K1902]

Epichlorohydrin C₃H₅ClO Colorless liquid, having a pungent odor similar to chloroform, specific gravity of 1.1801, and boiling point of 117°C. Purity: not less than 99%.

Ethanol See ethanol (95).

Ethanol, aldehyde-free Transfer 1000 mL of ethanol (95) to a glass-stoppered bottle, add the solution prepared by dissolving 2.5 g of lead (II) acetate trihydrate in 5 mL of water, and mix thoroughly. In a separate

container, dissolve 5 g of potassium hydroxide in 25 mL of warm ethanol (95), cool, and add this solution gently, without stirring, to the first solution. After 1 hour, shake this mixture vigorously, allow to stand overnight, decant the supernatant liquid, and distil the ethanol.

Ethanol, anhydrous See ethanol (99.5).

Ethanol, dilute To 1 volume of ethanol (95) add 1 volume of water. C_2H_5OH Content: 47.45 to 50.00 vol%

Ethanol (95) C_2H_5OH [K8102, Special class]

Ethanol (99.5) C_2H_5OH [K8101, Special class]

Ether See diethyl ether.

Ethyl iodine See iodoethane.

Ethylene bromide See 1,2-dibromoethane.

Ferric ammonium sulfate See ammonium iron (III) sulfate dodecahydrate.

Ferric chloride See iron (III) chloride hexahydrate.

Fluorescence reference standard (1) Weigh exactly 7 mg of fluorescent brightener (Fluorescent-24) in a brown volumetric flask, add water to make exactly 500 mL, and store protected from light.

To 5 mL of this solution, add 0.1 g of sodium chloride and water to make 50 mL, immerse a filter paper (8 cm × 15 cm) into the resultant solution for 1 hour with occasional inverting and air-dry it on a filter paper.

Fluorescence reference standard (2) Weigh exactly 7 mg of fluorescent brightener (Fluorescent-351) in a brown volumetric flask, add water to make exactly 500 mL, and store protected from light. To 0.35 mL of this solution, add 0.1 g of sodium chloride and water to make 50 mL, immerse a filter paper (8 cm × 15 cm) into the resultant solution for 1 hour with occasional inverting and air-dry it on a filter paper.

Fluorescent-24 $C_{40}H_{44}N_{12}Na_4O_{16}S_4$ Delayed-staining stilbene fluorescent brightener. Color index name: C.I. Fluorescent brightener-24 (C.I. No. 40650), Chemical name: Tetrasodium 4,4'-bis[[4-[bis(2-hydroxyethyl)amino]-6-[(3-sulphonato-phenyl)amino]-1,3,5-triazine-2-yl]amino]stilbene-2,2'-disulfonate (CAS No. 12224-02-1)

Fluorescent-351 $C_{28}H_{20}Na_2O_6S_2$ Stilbene fluorescent brightener Color index name: C.I. Fluorescent brightener-351 (FWA-5)(C.I.No.482200), Chemical name: Disodium 4,4'-Bis(2-sulfonatostyryl)biphenyl (CAS No. 27344-41-8)

Gauze Use medical gauze conforming to the requirements of Notification No. 0630001 of Secretary-General of Pharmaceutical and Food Safety Bureau, the MHLW dated June 30, 2005.

Glycerin $C_3H_8O_3$ [K8295, Special class] or [Concentrated Glycerin (JP monograph)]

Helium He, not less than 99.995 vol%

Heptane $CH_3(CH_2)_5CH_3$ [K9701, Special class]

n-Heptane See heptane.

Hexane for ultraviolet-visible spectrophotometry [K8848, Special class] When determining the absorbance of hexane as directed under Ultraviolet-visible Spectrophotometry, using water as the blank: not more than 0.10 at 220 nm and not more than 0.02 at 260 nm. It exhibits no absorption between 260 nm and 350 nm.

n-Hexane for ultraviolet-visible spectrophotometry See hexane for ultraviolet-visible spectrophotometry.

Hydrochloric acid HCl [K8180, Special class]

Hydrochloric acid, dilute Dilute 23.6 mL of hydrochloric acid with water to make 100 mL (10%).

0.1 mol/L Hydrochloric acid TS Dilute 100 mL of hydrochloric acid with water to make 1000 mL.

1 mol/L Hydrochloric acid TS Dilute 90 mL of hydrochloric acid with water to make 1000 mL.

Hydrogen peroxide (30) H₂O₂ [K8230, Hydrogen peroxide, Special class, Concentration: 30.0- 35.5%]

Hydrogen peroxide TS Dilute 1 volume of hydrogen peroxide (30) with 9 volumes of water. Prepare before use (3%).

Hydrogen peroxide water, strong See hydrogen peroxide (30).

Hydrogen sulfide H₂S Colorless, poisonous gas, heavier than air. It dissolves in water. Prepare by treating iron (II) sulfide with dilute sulfuric acid or dilute hydrochloric acid. Other sulfides yielding hydrogen sulfide with dilute acids may be used.

Hydroxylamine hydrochloride See hydroxylammonium chloride.

Hydroxylammonium chloride NH₂OH·HCl [K8201, Special class]

Hydroxylammonium hydrochloride See hydroxylammonium chloride.

Indium for thermal analysis Prepared for thermal analysis. Content: not less than 99.99%.

Iodine I [K8920, Iodine, Special class]

Iodine solution See iodine TS (0.05 mol/L).

Iodine TS Dissolve 14 g of iodine in 100 mL of a solution of potassium iodide (2 in 5), add 1 mL of dilute hydrochloric acid, and dilute with water to make 1000 mL (0.05 mol/L).

Storage: Preserve in light-resistant containers.

Iodoethane C₂H₅I A colorless to a dark-brown, clear liquid, having diethyl ether-like odor. Distilling range 71.0-72.5°C, not less than 94 vol%.

Iron (III) chloride hexahydrate FeCl₃·6H₂O [K8142, Special class]

Iron (II) sulfide FeS [K8948, for hydrogen sulfide development]

Isopropanol See 2-propanol.

Isopropyl alcohol See 2-propanol.

Isophorone diisocyanate C₁₂H₁₈N₂O₂ Colorless viscous liquid.

Isotonic sodium chloride solution [Isotonic Sodium Chloride Solution (JP monograph)]

Lead acetate See lead (II) acetate trihydrate.

Lead (II) acetate trihydrate Pb(CH₃COO)₂·3H₂O [K8374, Lead (II) acetate trihydrate, Special class]

Lead (II) acetate TS To 9.5 g of lead (II) acetate trihydrate add freshly boiled and cooled water to make 100 mL (0.25 mol/L). Preserve in tightly stoppered bottles.

Lead monoxide See lead (II) oxide.

Lead nitrate See lead (II) nitrate.

Lead (II) nitrate Pb(NO₃)₂ [K8563, Special class]

Lead (II) oxide PbO [K8090, Special class]

Lithium chloride LiCl White crystals or masses.

Identification: Perform the test as directed under Flame Coloration Test with this substance: a persistent red color develops.

Lithium chloride, anhydrous See lithium chloride.

Litmus paper, blue [K9071, Litmus paper, Blue litmus paper]

Litmus paper, red [K9071, Litmus paper, Red litmus paper]

Magnesium nitrate See magnesium nitrate hexahydrate.

Magnesium nitrate hexahydrate $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ [K8567, Special class]

Magnesium sulfate See magnesium sulfate heptahydrate.

Magnesium sulfate heptahydrate $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ [K8995, Special class]

Magnesium sulfate TS Dissolve 12 g of magnesium sulfate heptahydrate in water to make 100 mL (0.5 mol/L).

Methanol CH_3OH [K8891, Special class]

Methylene blue $\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S} \cdot 3\text{H}_2\text{O}$ [K 8897, Special class]

Methylene blue TS Dissolve 0.1g of methylene blue in 100 mL of ethanol, and filter if necessary.

Methylene blue TS, dilute Dilute 1 mL of methylene blue TS with water to make 100 mL.

Methylene iodide CH_2I_2 (another name: Diiodomethane) Yellow liquid.

Melting point: 6°C , boiling point: 181°C , specific gravity: d_{15}^{15} 3.3326

Methyl glycol acetate See acetic acid 2-methoxyethyl.

Methyl orange $\text{C}_{14}\text{H}_{14}\text{N}_3\text{NaO}_3\text{S}$ [K8893, Special class] Range of color change: pH (red) 3.1-4.4 (orange-yellow).

Methyl orange TS Dissolve 0.1 g of methyl orange in 100 mL of water, and filter if necessary.

Methyl red $\text{C}_{15}\text{H}_{15}\text{N}_3\text{O}_2$ [K8896, Special class] Range of color change: pH (red) 4.2-6.2 (yellow).

Methyl red TS Dissolve 0.1 g of methyl red in 100 mL of ethanol (95), and filter if necessary.

Nitric acid HNO_3 [K8541, Special class, Concentration: 69 - 70%, Density: about 1.42 g/mL]

Nitric acid, dilute Dilute 10.5 mL of nitric acid with water to make 100 mL (10%).

Nitrogen N_2 [Nitrogen (JP monograph)]

Octadecylsilanized silica gel for liquid chromatography Prepared for liquid chromatography

n-Octane C_8H_{18}

Specific gravity: d_4^{20} 0.700 – 0.705

Purity: Perform the test with 2 μL of this substance as directed under Gas Chromatography according to the conditions in the Assay under Hypromellose. Measure each peak area by the automatic integration method, and calculate the amount of n-octane by the area percentage method: not less than 99.0%.

Perchloroethylene [K 1521, Tetrachloroethylene]

Phenolphthalein $\text{C}_{20}\text{H}_{14}\text{O}_4$ [K8799, Special class] Range of color change: pH (colorless) 8.3-10.0 (red)

Phenolphthalein TS Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95).

Phloroglucin See phloroglucinol dehydrate.

Phloroglucinol dehydrate $\text{C}_6\text{H}_3(\text{OH})_3 \cdot 2\text{H}_2\text{O}$ White to pale yellow crystals or crystalline powder. Melting point: $215\text{-}219^\circ\text{C}$ (after drying), Loss on drying: 18.0-24.0% (1 g, 105°C , 1 hour)

Phosphorus (V) oxide P_2O_5 [K8342, Phosphorus (V) oxide, Special class]

Polyethylene glycol 20M for gas chromatography Prepared for gas chromatography.

Potassium bromide KBr [K8506, Special class]

Potassium bromide for infrared spectrophotometry Crush homocrystals of potassium bromide or potassium bromide, collect a powder passed through a No. 200 (75 μm) sieve, and dry at 120°C for 10 hours or at 500°C for 5 hours. Prepare tablets with this powder, and determine the infrared absorption spectrum: any

abnormal absorption does not appear.

Potassium bromate KBrO_3 [K8530, Special class]

Potassium bromate-potassium bromide TS Dissolve 1.4 g of potassium bromate and 8.1 g of potassium bromide in water to make 100 mL.

Potassium chloride KCl [K8121, Special class]

Potassium dihydrogen phosphate KH_2PO_4 [K9007, Potassium dihydrogen phosphate, Special class]

Potassium dihydrogen phosphate for pH determination KH_2PO_4 [K9007, Potassium dihydrogen

Potassium hydrogen phthalate for pH determination $\text{C}_6\text{H}_4(\text{COOK})(\text{COOH})$ [K8809, for pH standard solution]

Potassium hydroxide KOH [K8574, Special class]

Potassium hydroxide-ethanol TS Dissolve 10 g of potassium hydroxide in ethanol (95) to make 100 mL. Prepare before use.

Potassium iodide KI [K8913, Potassium iodide, Special class]

Potassium iodate (standard reagent) KIO_3 [K8005, Potassium iodate, Standard reagent for volumetric analysis]

Potassium iodide TS Dissolve 16.5 g of potassium iodide in water to make 100 mL. Preserve in light-resistant containers. Prepare before use (1 mol/L).

Potassium permanganate KMnO_4 [K8247, Special class]

Potassium tetraoxalate for pH determination See potassium trihydrogen dioxalate dihydrate for pH determination.

Potassium trihydrogen dioxalate dihydrate for pH determination $\text{KH}_3(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$ [K8474, Potassium trihydrogen dioxalate dihydrate for pH determination]

2-Propanol $(\text{CH}_3)_2\text{CHOH}$ [K8839, Special class]

Pyridine $\text{C}_5\text{H}_5\text{N}$ [K8777, Special class]

Red litmus paper See litmus paper, red.

Silica gel An amorphous, partly hydrated silicic acid occurring in glassy granules of various sizes. When used as a desiccant, it is frequently coated with a substance that changes color when the capacity to absorb water is exhausted. Such colored products may be regenerated by being heated at 110°C until the gel assumes the original color.

Loss on ignition: Not more than 6% (2 g, $950 \pm 50^\circ\text{C}$)

Water absorption: Not less than 31%. Weigh accurately about 10 g of this substance, and allow to stand for 24 hours in a closed container in which the atmosphere is maintained at 80% relative humidity with sulfuric acid having a specific gravity of 1.19. Weigh again, and calculate the increase in mass.

Siliceous earth [K8330, Siliceous earth, First class]

Siliceous earth for gas chromatography Prepared for gas chromatography.

Silicone oil Colorless clear liquid, having no odor. Viscosity 50-100 mm^2/s

Silver diethyldithiocarbamate See silver N, N-diethyldithiocarbamate.

Silver N,N-diethyldithiocarbamate $\text{C}_5\text{H}_{10}\text{AgNS}_2$ [K9512, Special class]

Silver nitrate AgNO_3 [K8550, Special class]

Silver nitrate TS Dissolve 17.5 g of silver nitrate in water to make 1000 mL (0.1 mol/L). Preserve in light-resistant containers.

Soda lime [K8603, for carbon dioxide absorption]

Sodium acetate See sodium acetate trihydrate.

Sodium acetate trihydrate $\text{CH}_3\text{COONa}\cdot 3\text{H}_2\text{O}$ [K8371, Special class]

Sodium borate for pH determination See sodium tetraborate decahydrate for pH determination.

Sodium bromide NaBr [K8514, Special class]

Sodium carbonate, anhydrous Na_2CO_3 [K8625, Sodium carbonate, Special class]

Sodium carbonate for pH determination Na_2CO_3 [K8625, for pH standard solution]

Sodium carbonate (standard reagent) Na_2CO_3 [K8005, Sodium carbonate, Standard reagent for volumetric analysis]

Sodium chloride NaCl [K8150, Special class]

Sodium hydrogen carbonate for pH determination NaHCO_3 [K8622, for pH standard solution]

Sodium hydroxide NaOH [K8576, Special class]

Sodium hydroxide TS Dissolve 4.3 g of sodium hydroxide in water to make 100 mL (1 mol/L). Preserve in polyethylene bottles.

Sodium oxalate (standard reagent) $\text{C}_2\text{O}_4\text{Na}_2$ [K8005, Sodium oxalate, Standard reagent for volumetric analysis] or [K8528, Sodium oxalate, Special class]

Sodium sulfate, anhydrous Na_2SO_4 [K8987, Sodium sulfate, Special class]

Sodium sulfide enneahydrate $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ [K8949, Special class]

Sodium sulfide TS Dissolve 5 g of sodium sulfide enneahydrate in a mixture of 10 mL of water and 30 mL of glycerin. Or dissolve 5 g of sodium hydroxide in a mixture of 30 mL of water and 90 mL of glycerin, saturate a half volume of this solution with hydrogen sulfide, while cooling, and mix with the remaining half. Preserve in well-filled, light-resistant bottles. Use within 3 months.

Sodium tetraborate decahydrate for pH determination [K8866, Sodium tetraborate decahydrate for pH standard solution]

Sodium thiosulfate See sodium thiosulfate pentahydrate.

Sodium thiosulfate pentahydrate $\text{Na}_2\text{S}_2\text{O}_3\cdot 5\text{H}_2\text{O}$ [K8637, Special class]

Stannous chloride See tin (II) chloride dihydrate.

Stannous chloride TS, acidic See tin (II) chloride TS, acidic.

Starch [K8658, Starch, Special class]

Starch TS Triturate 1 g of starch with 10 mL of cold water, and pour the mixture slowly, with constant stirring, into 200 mL of boiling water. Boil the mixture until a thin, translucent fluid is obtained. Allow to settle, and use the supernatant liquid. Prepare before use.

Strong ammonia water See ammonia solution (28).

Strong ammonium water See ammonia solution (28).

Strong hydrogen peroxide water See hydrogen peroxide (30).

Styrene C_8H_8 Clear and colorless liquid.

Specific gravity: 0.902-0.910.

Purity Perform the test with 1 μL of this substance as directed under Gas Chromatography according to the following conditions. Measure each peak area by the automatic integration method and calculate the amount of styrene by the area percentage method: it shows the purity of not less than 99%.

Operating conditions

Detector: Thermal conductivity detector

Column: A glass column about 3 mm in inside diameter and about 2 m in length, packed with siliceous earth for gas chromatography (180 to 250 μm) coated with polyethylene glycol 20 M for gas chromatography in 10%

Column temperature: A constant temperature of about 100°C

Temperature of sample vaporization chamber: A constant temperature of about 150°C

Carrier gas: Helium

Flow rate: Adjust the flow rate so that the retention time of styrene is about 10 minutes.

Time span of measurement: About twice as long as the retention time of styrene

Sulfamic acid (standard reagent) See amidosulfuric acid (standard reagent).

Sulfanilamide reference standard for melting point determination [JP Sulfanilamide reference standard for melting point determination]

Sulfapyridine reference standard for melting point determination [JP Sulfapyridine reference standard for melting point determination]

Sulfuric acid H_2SO_4 [K8951, Special class]

Sulfuric acid (94.5-95.5%) See sulfuric acid for readily carbonizable substances.

Sulfuric acid, dilute Cautiously add 5.7 mL of sulfuric acid to 10 mL of water, cool, and dilute with water to make 100 mL (10%).

Sulfuric acid for readily carbonizable substances To sulfuric acid, the content of which has previously been determined by the following method, add water cautiously, and adjust the final concentration to 94.5% to 95.5% of sulfuric acid (H_2SO_4). When the concentration is changed owing to absorption of water during storage, prepare freshly.

Assay: Weigh accurately about 2 g of sulfuric acid in a glass-stoppered flask rapidly, add 30 mL of water, cool, and titrate the solution with 1 mol/L sodium hydroxide VS (indicator: 2 to 3 drops of bromothymol blue TS).

Each mL of 1 mol/L sodium hydroxide VS = 49.04 mg H_2SO_4

Tetrahydrofuran $\text{CH}_2(\text{CH}_2)_2\text{CH}_2\text{O}$ [K9705, Special class]

Tetrahydrofuran for gas chromatography Use tetrahydrofuran prepared by distilling with iron (II) sulfate heptahydrate. Preserve in containers, in which the air has been displaced by nitrogen, in a dark, cold place.

Tin (II) chloride dihydrate $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ [K8136, Special class]

Tin (II) chloride TS, acidic Dissolve 8 g of Tin (II) chloride dihydrate in 500 mL of hydrochloric acid.

Preserve in glass-stoppered bottles. Use within 3 months.

Tin for thermal analysis [K8580, Tin, Special class, Content: not less than 99.99%]

Toluene $\text{C}_6\text{H}_5\text{CH}_3$ [K8680, Special class]

Vanillin reference standard for melting point determination [JP Vanillin reference standard for melting point determination]

Vinyl chloride $\text{C}_2\text{H}_3\text{Cl}$ Colorless gas. Boiling point: -14°C, Melting point: -160°C

Xylene $\text{C}_6\text{H}_4(\text{CH}_3)_2$ [K8271, First class]

Zinc for arsenic analysis Zn [K 8012, for arsenic analysis] Use granules of about 800 μm .

Zinc, arsenic-free See zinc for arsenic analysis.

Zinc chloride ZnCl_2 [K8111, Special class]

(5) Measuring Instruments and Appliances

Balances and weights

- (1) Chemical balances Use balances readable to the extent of 0.1 mg.
- (2) Semimicrobalances Use balances readable to the extent of 0.01 mg.
- (3) Microbalances Use balances readable to the extent of 0.001 mg.
- (4) Weights Use calibrated weights.

Filter paper

Use the filter paper conforming to the following specifications. A filter paper without specification means that for qualitative analysis. Store protected from contamination with gas, etc.

Filter paper for qualitative analysis

Use the filter papers conforming to the specifications for filter papers for qualitative analysis (for chemical analysis) provided in P3801 of the JIS.

Filter papers for quantitative analysis

Use the filter papers conforming to the specifications for filter papers for quantitative analysis (for chemical analysis) provided in P3801 of the JIS.

Gas flowmeters for gas chromatography

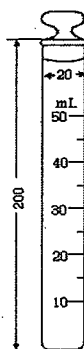
Use the flowmeter provided in K1518 of the JIS.

Micro-syringes for gas chromatography and liquid chromatography

Use commercial micro-syringes for gas chromatography.

Nessler tubes

Use colorless, glass-stoppered cylinders 1.0 to 1.5 mm in thickness, made of hard glass as shown in the figure below. The difference of the height of the graduation line of 50 mL from the bottom among cylinders does not exceed 2 mm.



(The figures are in mm.)

Sieves

Use the sieves provided in Z8801 of the JIS.

Use the sieve number or nominal size (μm) as the designation.

Bulb length (mm)	12-15	12-15	12-15	12-15	12-15	12-15
Distance from bottom of bulb to graduation at the lowest temperature (mm)	75-90	75-90	75-90	75-90	75-90	75-90
Distance from top of thermometer to graduation at the highest temperature (mm)	35-50	35-50	35-50	35-50	35-50	35-50
Distance from bottom of bulb to immersion line(mm)	60	60	60	60	60	60
From of top of thermometer	loop	loop	loop	loop	loop	loop
Maximum scale error at any point	0.2°C	0.2°C	0.2°C	0.2°C	0.2°C	0.2°C

For the No. 1, No. 2 and No. 6 thermometers, the quality of the glass shall be of Jena 16III or higher, and for No. 4, No. 5 and No. 6, Jena 59III or higher.

Thermometers for low pour-point

Use the thermometers provided in B7410 of the JIS.

Thermometers for high pour-point

Use the thermometers provided in B7410 of the JIS.

Volumetric measures for chemical use

Use volumetric flasks, transfer pipets, burettes and measuring cylinders provided in R3505 of the JIS.

<3. Monographs>

Part 1 - Materials

Ionomer Resin

Definition

Ionomer Resin is a copolymer resin, partially cross-linked with sodium hydroxide or zinc oxide, obtained by copolymerizing ethylene and acrylic acid or ethylene and methacrylic acid.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 3440 cm^{-1} , between 3000 cm^{-1} and 2900 cm^{-1} , 1378 cm^{-1} , 1190 cm^{-1} , 1140 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.93-0.99

Melting point: 80-100°C

Purity

(1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

(2) Heavy metals: Not more than 20 ppm (Method 2)

(3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Alkyl Acrylate-Vinyl Acetate Copolymer Emulsion (2)

Definition

Alkyl Acrylate-Vinyl Acetate Copolymer Emulsion (2) is a copolymer emulsion mainly consisting of more than one component in alkyl acrylate ($C_1 - C_4$, C_8 and C_{12}) and alkyl methacrylate ($C_1 - C_4$, C_8 and C_{12}), and vinyl acetate.

Description

It is a milky white liquid and it has a faint, characteristic odor.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about between 2940 cm^{-1} and 2900 cm^{-1} , 1730 cm^{-1} (ester), 1450 cm^{-1} , 1240 cm^{-1} , 1020 cm^{-1} and 720 cm^{-1} .

pH: 6.0-8.5

Purity

(1) Heavy metals: Not more than 20 ppm (Method 2)

(2) Arsenic: Not more than 2 ppm (Method 2)

Partial Potassium Salt of Polyacrylic Acid

Definition

Partial Potassium Salt of Polyacrylic Acid is a water-absorbing resin consisting of slightly cross-linked partial

potassium salt of polyacrylic acid as a principal component.

Description

- (1) It occurs as a white powder and it is practically odorless.
- (2) It swells with water but is practically insoluble in water.
- (3) Melting point: Not less than 200°C (with decomposition)

Identification

- (1) To 1.0 g of this substance, add 100 mL of water, stir and allow to stand for 10 minutes: the solution becomes gelatinous.
- (2) To 10 g of the gelatinous substance (1), add 1 mL of calcium chloride TS and shake: a white precipitate is produced.
- (3) To 10 g of the gelatinous substance (1), add 1 mL of magnesium sulfate TS and shake: a white precipitate is produced.
- (4) To 10 g of the gelatinous substance (1), add 1 mL of cobalt chloride solution (1 in 25), add 2 or 3 drops of ammonium chloride TS and shake: a light red precipitate is produced. Dry the precipitate: a purple color develops.
- (5) Ignite this substance to incinerate and perform the test with the residue as directed under Flame Coloration Test: it responds to the Qualitative Test for potassium.

Purity

- (1) Coloring matter

Immerse this substance in ethanol not less than 10 times the mass of this substance, stir for 10 minutes and filter: the filtrate is a colorless clear liquid.

- (2) Acidity or alkalinity

To 1.0 g of this substance, add 500 mL of freshly boiled and cooled water and allow to cool. To 25 mL of this solution, add 3 drops of phenolphthalein TS: no red color develops. Separately, to 25 mL of the solution, add 1 drop of methyl orange TS: a yellow color develops.

- (3) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows no marked fluorescence.

- (4) Heavy metals: Not more than 20 ppm (Method 2)

- (5) Acrylic acid

Method 1

To 5.0 g of this substance, add exactly 10 mL of methanol, shake for 4 hours, allow to stand and use the supernatant liquid as the sample solution. Separately, take 0.010 g of the acrylic acid reference standard, dissolve in methanol to make exactly 200 mL and use the solution as the standard solution. Perform the test with 5 µL each of the sample solution and standard solution as directed under Gas Chromatography. Determine the peak heights, H_t and H_s, of acrylic acid of respective solutions: H_t is not higher than H_s.

Method 2

To 1.0 g of this substance, add 250 mL of saline, stir for 2 hours, filter, and use the resultant solution as

the sample solution. Separately, take 0.20 g of acrylic acid reference standard, dissolve in saline to make exactly 100 mL. To 1 mL of the solution, add saline to make exactly 250 mL and use as the standard solution. Perform the test with 20 μ L each of the sample solution and the standard solution as directed under Liquid Chromatography. Determine the peak heights, H_t and H_s , of acrylic acid of respective solutions: H_t is not higher than H_s .

Loss on drying: Not more than 15% (2.0 g, 105°C, 3 hours)

Residue on ignition: Not more than 76% (Method 1)

Absorbency

Take 1.0 g of this substance in a nylon woven fabric (10 cm in width, 20 cm in length, 255 mesh), immerse in 1000 mL of saline for 1 hour, allow to stand for 10 minutes, remove excessive water and determine absorbency: it absorbs not less than 10 times its weight.

Note) Identify acrylic acid according to Method 1 or Method 2.

Partial Sodium Salt of Polyacrylic Acid

Definition

Partial Sodium Salt of Polyacrylic Acid is a water-absorbing resin consisting of slightly cross-linked partial sodium salt of polyacrylic acid as a principal component.

Description

- (1) It occurs as a white powder and it is practically odorless.
- (2) It swells with water but is practically insoluble in water.
- (3) Melting point: Not less than 200°C (with decomposition)

Identification

- (1) To 1.0 g of this substance, add 100 mL of water, stir and allow to stand for 10 minutes: the solution becomes gelatinous.
- (2) To 10 g of the gelatinous substance (1), add 1 mL of calcium chloride TS and shake: a white precipitate is produced.
- (3) To 10 g of the gelatinous substance (1), add 1 mL of magnesium sulfate TS and shake: a white precipitate is produced.
- (4) To 10 g of the gelatinous substance (1), add 1 mL of cobalt chloride solution (1 in 25), add 2 or 3 drops of ammonium chloride TS and shake: a light red precipitate is produced. Dry the precipitate: a purple color develops.
- (5) Ignite this substance to incinerate and perform the test with the residue as directed under the Flame Coloration Test: it responds to the Qualitative Test for sodium.

Purity

- (1) Coloring matter

Immerse this substance in ethanol not less than 10 times the mass of this substance, stir for 10 minutes and filter: the filtrate is a colorless clear liquid.

- (2) Acidity or alkalinity

To 1.0 g of this substance, add 500 mL of freshly boiled and cooled water and allow to cool. To 25 mL of this solution, add 3 drops of phenolphthalein TS: no red color develops. Separately, to 25 mL of the solution, add 1 drop of methyl orange TS: a yellow color develops.

(3) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows no marked fluorescence.

(4) Heavy metals: Not more than 20 ppm (Method 2)

(5) Acrylic acid

Method 1

To 5.0 g of this substance, add exactly 10 mL of methanol, shake for 4 hours, allow to stand and use the supernatant liquid as the sample solution. Separately, take 0.010 g of the acrylic acid reference standard, dissolve in methanol to make exactly 200 mL and use the solution as the standard solution. Perform the test with 5 μ L each of the sample solution and standard solution as directed under Gas Chromatography. Determine the peak heights, H_t and H_s , of acrylic acid of respective solutions: H_t is not higher than H_s .

Method 2

To 1.0 g of this substance, add 250 mL of saline, stir for 2 hours, filter, and use the resultant solution as the sample solution. Separately, take 0.20 g of acrylic acid reference standard, dissolve in saline to make exactly 100 mL. To 1 mL of the solution, add saline to make exactly 250 mL and use as the standard solution. Perform the test with 20 μ L each of the sample solution and the standard solution as directed under Liquid Chromatography. Determine the peak heights, H_t and H_s , of acrylic acid of respective solutions: H_t is not higher than H_s .

Loss on drying: Not more than 15% (2.0 g, 105°C, 3 hours)

Residue on ignition: Not more than 76% (Method 1)

Absorbency

Take 1.0 g of this substance in a nylon woven fabric (10 cm in width, 20 cm in length, 255 mesh), immerse in 1000 mL of saline for 1 hour, allow to stand for 10 minutes, remove excessive water and determine absorbency: it absorbs more than 10 times its weight.

Note) Identify acrylic acid according to Method 1 or Method 2.

Acetate Fiber

Definition

Acetate Fiber is a cellulose fiber obtained by partially saponifying acetylated cellulose.

Description

It occurs as colorless to light yellow fibrous substances, and it is odorless.

Identification

- (1) Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 3440 cm^{-1} , 2920 cm^{-1} , 1760 cm^{-1} , 1360 cm^{-1} , 1240 cm^{-1} , 1040 cm^{-1} and 900 cm^{-1} .

- (2) When burned, it softens, shrinks, and burns gradually. Solid and black mass remains, but it can be crushed with a hand easily.

Specific gravity: 1.30-1.32

Melting point: 260°C

Purity

(1) Heavy metals: Not more than 20 ppm (Method 2)

(2) Arsenic: Not more than 2 ppm (Method 2)

Loss on drying: Not more than 8.0% (2.0 g, 105°C, 3 hours)

Residue on ignition: Not more than 2.5% (Method 2)

Alkyl Ketene Dimer Emulsion

Definition

Alkyl Ketene Dimer Emulsion is an emulsion obtained by the emulsification of alkyl ketene dimer.

Description

It is a white liquid, and it is odorless or has a faint, characteristic odor.

Identification

Determine the infrared absorption spectrum of this substance, previously dried at 105°C for 3 hours, as directed in the potassium bromide disk method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about between 3000 cm^{-1} and 2800 cm^{-1} , 1850 cm^{-1} , 1720 cm^{-1} and 1470 cm^{-1} .

pH: 3.0-4.5

Purity

(1) Heavy metals: Not more than 10 ppm (Method 2)

(2) Arsenic: Not more than 2 ppm (Method 2)

Alkenyl Succinic Anhydride Solution

Definition

Alkenyl Succinic Anhydride Solution is obtained by the maleinization of alkenyl succinic anhydride and maleic anhydride. It is saponified with potassium hydroxide.

Description

It is a red brown to light red brown liquid, and it has a faint, characteristic odor.

Identification

Determine the infrared absorption spectrum of this substance, previously dried at 105°C for about 2 hours, as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about between 3000 cm^{-1} and 2800 cm^{-1} , 1570 cm^{-1} , and 1410 cm^{-1} .

pH: 7.0-9.5

Purity

(1) Heavy metals: Not more than 10 ppm (Method 2)

(2) Arsenic: Not more than 2 ppm (Method 2)

Sulfur

Definition

Sulfur, when dried, contains not less than 99.5% of sulfur (S).

Description

It occurs as a light yellow, fine, amorphous or crystalline powder, and it is odorless.

Identification

Ignite this substance: it burns with a blue flame and gives a characteristic odor of sulfur dioxide.

Purity

(1) Acidity or alkalinity

To 2.0 g of this substance, add 10 mL of water, shake and filter: the solution is neutral.

(2) Arsenic

To 0.2 g of this substance, add 10 mL of ammonia TS, stir for 3 hours, filter, evaporate the filtrate on a water bath to dryness, add 1 mL of nitric acid and evaporate the solution to dryness again. To the residue, add 5 mL of water, and perform the test using this solution as the sample solution: not more than 10 ppm.

Loss on drying: Not more than 1.0% (1.0 g, silica gel, 4 hours)

Residue on ignition: Not more than 0.3% (Method 1)

Assay

Weigh accurately about 1 g of this substance, previously dried, add 50 mL of potassium hydroxide-ethanol TS, dissolve by boiling, allow to cool and add water to make 250 mL. To 25 mL of this solution, add 50 mL of hydrogen peroxide TS and heat on a water bath for 1 hour. Acidify with dilute hydrochloric acid, add 200 mL of water and add dropwise hot barium chloride TS while boiling until no more precipitate is produced. Heat the mixture on a water bath for 1 hour, filter the precipitate, and wash thoroughly with water. Ignite the precipitate to constant mass, weigh the residue as barium sulfate (BaSO_4 233.40). Perform a blank determination in the same manner, and make any necessary correction.

$$\text{Amount (mg) of S} = \text{amount (mg) of barium sulfate (BaSO}_4) \times 0.1374$$

Urethane Fiber

Definition

Urethane Fiber is a fiber obtained by the reaction of polytetramethylene ether glycol with diphenylmethane diisocyanate, followed by dilution with dimethylacetamide solvent and polymerization with amine compound.

Description

It occurs as a white elastomer and it is odorless.

Identification

Dissolve this substance in dimethylacetamide (1 in 5), and determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about between 3500 cm^{-1} and 3250 cm^{-1} , between 1750 cm^{-1} and 1690 cm^{-1} , between 1575 cm^{-1} and 1500 cm^{-1} , and 1100 cm^{-1} (ether bond).

Melting point: Not less than 225°C (with decomposition)

Purity

(1) Coloring matter

Immerse 10 g of this substance in 100 mL of ethanol, allow to cool, stir for about 10 minutes and filter. Transfer 50 mL of the filtrate into a Nessler tube and observe downward: the filtrate is colorless and clear.

(2) Acidity or alkalinity

To 1 g of this substance, add 300 mL of freshly boiled and cooled water and allow to cool. To 25 mL of the solution, add 3 drops of phenolphthalein TS: no red color develops. Separately, to 25 mL of the same solution, add 1 drop of methyl orange TS: no red color develops.

(3) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows neither marked fluorescence nor fluorescence by contamination.

(4) Residual isocyanate

Weigh accurately 0.3 g of this substance, add 3 mL of methanol, extract at 70°C for 3 hours, and use the extract as the sample solution. Separately, to 0.02 g of diphenylmethane diisocyanate, add methanol to make exactly 100 mL. To 1 mL of this solution, add methanol to make 10 mL, and use this solution as the standard solution. Perform the test with 5 µL each of the sample solution and the standard solution as directed under Liquid Chromatography according to the following conditions: the peak area of the product obtained by the reaction of isocyanate and methanol from the sample solution is not larger than the peak area of the product obtained by the reaction of isocyanate and methanol from the standard solution.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 246 nm)

Column: A stainless steel column, 4.6 mm in inside diameter and 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography

Mobile phase: To 50 mL of acetonitrile, add 50 mL of water.

Flow rate: 1.0 mL/min (Adjust it so that the retention time of the product obtained by the reaction of isocyanate and methanol is about 5 minutes)

(5) Heavy metals: Not more than 20 ppm (Method 2)

Residue on ignition: Not more than 5% (Method 3)

Urethane Film

Definition

Urethane Film is a film obtained by the extrusion molding of molten polyurethane.

Description

It occurs as a translucent elastomer and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared

Spectrophotometry: it exhibits absorption at the wave numbers of about between 3400 cm^{-1} and 3300 cm^{-1} , 1740 cm^{-1} , 1530 cm^{-1} , and between 1170 cm^{-1} and 1070 cm^{-1} .

Purity

(1) Coloring matter

Immerse 10 g of this substance in 100 mL of freshly boiled and cooled water, stir and filter. Transfer 50 mL of the filtrate into a Nessler tube and observe downward: the filtrate is almost colorless.

(2) Acidity or alkalinity

Transfer 25 mL of the filtrate (1) into a test tube 15 mm in inside diameter and add 3 drops of phenolphthalein TS: no red color develops. Separately, to 25 mL of the same solution, add 1 drop of methyl orange TS: no red color develops.

(3) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows neither marked fluorescence nor fluorescence by contamination.

(4) Heavy metals: Not more than 20 ppm (Method 2)

Urethane Foam

Definition

Urethane Foam is a soft polyester polyol urethane foam.

Description

It occurs as a white porous elastomer and it is odorless.

Identification

(1) Confirmation of urethane bond

Take about 0.2 g of this substance in a test tube and stopper loosely with absorbent cotton. Decompose this substance by heating the test tube over a low flame, immerse the absorbent cotton in a solution of p-dimethylaminobenzaldehyde in methanol (1 in 100) and acidify with 1 drop of dilute hydrochloric acid: a bright yellow color develops.

(2) Confirmation of ester bond

Take about 0.05 g of this substance in a test tube, add several drops of a solution of potassium hydroxide in methanol (11.2 in 100) and several drops of a saturated solution of hydroxylamine hydrochloride in methanol. Add several drops of phenolphthalein TS and confirm this mixture is alkaline. Heat the mixture on a water bath for 20-40 seconds, allow to cool about 1 minute and acidify with 1 mol/L hydrochloric acid TS. To this solution, add ferric chloride solution (2 in 100): a purple color develops.

pH

To 1.0 g of this substance, add 100 mL of freshly boiled and cooled water, press out and determine the pH as directed under pH Determination: it is between 6.0 and 7.5.

Melting point: 225-240°C (Method 1, with decomposition)

Purity

(1) Heavy metals: Not more than 20 ppm (Method 2)

(2) Unreacted monomer

To 1.0 g of this substance, previously dried, add benzene solution of dibenzylamine (1 in 100,000) to make exactly 100 mL, and heat on a hot bath for 1 hour. After cooling, press to collect the extract. Immerse the extract in 50 mL of benzene, and press to collect the extract, and repeat this three more times. Evaporate the extract to dryness under reduced pressure, add 1 mL of methanol, and use this solution as the sample solution. Separately, to 0.020 g of isophorone diisocyanate, add benzene to make exactly 100 mL. To 1 mL of the solution, add benzene solution of dibenzylamine (1 in 100,000) to make exactly 100 mL, and heat on a hot bath for 1 hour. After cooling, evaporate it to dryness under reduced pressure, add 1 mL of methanol, and use this solution as the standard solution. Perform the test with 20 μ L each of the sample solution and standard solution as directed under Liquid Chromatography according to the following conditions. Determine the peak heights, H_t and H_s , of isophorone diisocyanate of respective solutions: H_t is not higher than H_s .

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 265 nm)

Column: A stainless steel column, 4.6 mm in inside diameter and 150 mm in length, packed with 6- μ m silica gel chemically bounded with an octadecyl group

Mobile phase: To 85 mL of methanol, add 15 mL of water.

Flow rate: 0.7 mL/min (Adjust the flow rate so that the retention time of the product obtained by the reaction of isophorone diisocyanate and dibenzylamine is about 10 minutes)

Detection sensitivity: Adjust it so that the peak height of isophorone diisocyanate obtained from 20 μ L of the standard solution is 5 to 10 mm.

Residue on ignition: 0.2% (Method 1)

Ester Gum

Definition

Ester Gum is an ester compound of the derivatives of rosin or its polymer.

Description

It occurs as a light yellow to light brown glass mass or clear viscous liquid, and it is odorless or has a faint, characteristic odor.

Identification

- (1) To 0.1 g of this substance, add 10 mL of acetic anhydride, dissolve by heating in a water bath, allow to cool and add 1 drop of sulfuric acid: a purple-red color develops.
- (2) To 1 g of this substance, add 5 mL of sodium hydroxide solution (1 in 25) and 5 mL of water and shake vigorously: a white to light yellow turbidity and a lasting foam is produced.

Purity

- (1) Clarity of solution

Dissolve 10 g of this substance in 10 mL of toluene at 70-75°C, filter while warming and allow the

solution to stand for 24 hours: the solution is clear.

- (2) Acid value: Not more than 18.0 (Method 1)

Weigh accurately about 3 g of this substance, dissolve in 50 mL of a mixture of benzene and ethanol (2:1), and perform the test with this solution as the sample solution.

- (3) Heavy metals: Not more than 40 ppm (0.50 g, Method 2, Standard Lead Solution 2.0 mL)

- (4) Arsenic: Not more than 4 ppm (0.25 g, Method 2)

Residue on ignition: Not more than 0.1% (2.0 g, Method 1)

Ethylene-Ethyl Acrylate Copolymer (EEA)

Definition

Ethylene-Ethyl Acrylate Copolymer (EEA) is an ethylene-ethyl acrylate copolymer resin obtained by copolymerizing of ethylene and ethyl acrylate.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about between 3000 cm^{-1} and 2900 cm^{-1} , 1740 cm^{-1} , 1460 cm^{-1} , 1378 cm^{-1} , 1160 cm^{-1} , 1140 cm^{-1} , 730 cm^{-1} , and 720 cm^{-1} .

Specific gravity: 0.91-0.98

Melting point: 60-100°C

Purity

- (1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

- (2) Heavy metals: Not more than 20 ppm (Method 2)

- (3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Ethylene-Acrylic Acid Copolymer (EAA)

Definition

Ethylene-Acrylic Acid Copolymer (EAA) is an ethylene-acrylic acid copolymer resin obtained by copolymerizing ethylene and acrylic acid.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2870 cm^{-1} , 1379 cm^{-1} , 730 cm^{-1} , 722 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.94-0.98

Melting point: 80-100°C

Purity

(1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

(2) Heavy metals: Not more than 20 ppm (Method 2)

(3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Ethylene-Octene-1 Copolymer

Definition

Ethylene-Octene-1 Copolymer is an ethylene-octene-1 copolymer resin obtained by copolymerizing ethylene and octene-1.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2870 cm^{-1} , 1460 cm^{-1} , 1378 cm^{-1} , 899 cm^{-1} , 730 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.85-0.94

Melting point: 115-130°C

Purity

(1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

(2) Heavy metals: Not more than 20 ppm (Method 2)

(3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Ethylene-Vinyl Acetate Copolymer (EVA) (1)

Definition

Ethylene-Vinyl Acetate Copolymer (EVA) (1) is an ethylene-vinyl acetate copolymer obtained by heating and polymerizing ethylene and vinyl acetate under high pressure using organic peroxide as a reaction initiator.

Description

(1) It occurs as a milky white pelleted or powdered solid.

(2) It is soluble in toluene and xylene, and practically insoluble in water.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 1740 cm^{-1} and 1470 cm^{-1} .

Purity

(1) Clarity of solution

Dissolve 1 g of this substance in 100 mL of hot xylene: the solution is clear.

(2) Heavy metals: Not more than 10 ppm (Method 2)

Loss on drying: Not more than 0.5% (1.0 g, 80°C, 4 hours)

Residue on ignition: Not more than 0.5% (30 g, 900°C, 90 minutes)

Ethylene-Vinyl Acetate Copolymer (EVA) (2)

Definition

Ethylene-Vinyl Acetate Copolymer (EVA) (2) is a copolymer resin obtained by radical reaction of ethylene and vinyl acetate (3% to 40%).

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2920 cm^{-1} , 2850 cm^{-1} , 1735 cm^{-1} , 1470 cm^{-1} , 1370 cm^{-1} , 1235 cm^{-1} , 1020 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.91-0.98

Melting point: 60-100°C

Purity

(1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

(2) Heavy metals: Not more than 20 ppm (Method 2)

(3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Ethylene-Vinyl Acetate Copolymer Emulsion

Definition

Ethylene-Vinyl Acetate Copolymer Emulsion is a copolymer emulsion obtained by the emulsion polymerization of ethylene and vinyl acetate under medium pressure.

Description

It is a milky white liquid, and it is odorless or has a faint, characteristic odor.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about between 2900 cm^{-1} and 2850 cm^{-1} , 1740 cm^{-1} , 1240 cm^{-1} , and 610 cm^{-1} .

pH: 4.0-7.0

Purity

(1) Heavy metals: Not more than 10 ppm (Method 2)

(2) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.4% (Method 1)

Ethylene-Vinyl Acetate/Polypropylene Bicomponent Fiber

Definition

Ethylene-Vinyl Acetate/Polypropylene Bicomponent Fiber is a polypropylene-core/ethylene-vinyl acetate copolymer-sheath or side-by-side bicomponent fiber.

Description

It occurs as colorless to white fibrous substances, and it is odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2880 cm^{-1} , 2820 cm^{-1} , 1740 cm^{-1} , 1450 cm^{-1} , 1360 cm^{-1} , 1240 cm^{-1} , 1010 cm^{-1} , 720 cm^{-1} and 600 cm^{-1} .

Specific gravity: 0.93-0.98

Melting point: 90-110°C

Purity

(1) Heavy metals: Not more than 20 ppm (Method 2)

(2) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 2.5% (Method 2)

Ethylene-Butene Copolymer (EBR)

Definition

Ethylene-Butene Copolymer (EBR) is an ethylene-butene copolymer resin obtained by copolymerizing ethylene and butene.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2870 cm^{-1} , 1379 cm^{-1} , 772 cm^{-1} , 730 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.85-0.90

Melting point: 120-130°C

Purity

(1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

(2) Heavy metals: Not more than 20 ppm (Method 2)

(3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Ethylene-Propylene Copolymer (EPR)

Definition

Ethylene-Propylene Copolymer (EPR) is an ethylene-propylene copolymer resin obtained by copolymerizing ethylene and propylene.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2870 cm^{-1} , 1735 cm^{-1} , 1460 cm^{-1} , 1377 cm^{-1} , 1150 cm^{-1} , 937 cm^{-1} , 730 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.85-0.88

Melting point: 120-130°C

Purity

(1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

(2) Heavy metals: Not more than 20 ppm (Method 2)

(3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Ethylene-Pentene-1 Copolymer

Definition

Ethylene-Pentene-1 Copolymer is an ethylene-pentene-1 copolymer resin obtained by copolymerizing ethylene and pentene-1.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2870 cm^{-1} , 1460 cm^{-1} , 1378 cm^{-1} , 894 cm^{-1} , 730 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.85-0.94

Melting point: 120-130°C

Purity

(1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

(2) Heavy metals: Not more than 20 ppm (Method 2)

(3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Ethylene-Methacrylic Acid Copolymer (EMAA)

Definition

Ethylene-Methacrylic Acid Copolymer (EMAA) is an ethylene-methacrylic acid copolymer resin obtained by copolymerizing ethylene and methacrylic acid.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2870 cm^{-1} , 1710 cm^{-1} , 1460 cm^{-1} , 730 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.92-0.98

Melting point: 80-105°C

Purity

(1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

(2) Heavy metals: Not more than 20 ppm (Method 2)

(3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Ethylene-Methyl Methacrylate Copolymer (EMMA)

Definition

Ethylene-Methyl Methacrylate Copolymer (EMMA) is an ethylene-methyl methacrylate copolymer resin obtained by copolymerizing ethylene and methyl methacrylate.

Description

It occurs as translucent powder or granules, and is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 3440 cm^{-1} , between 3000 cm^{-1} and 2900 cm^{-1} , 1378 cm^{-1} , 1190 cm^{-1} , 1140 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.92-0.95

Melting point: 60-110°C

Purity

(1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

(2) Heavy metals: Not more than 20 ppm (Method 2)

(3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Ethylene-4-Methylpentene-1 Copolymer

Definition

Ethylene-4-Methylpentene-1 Copolymer is an ethylene-4-methylpentene-1 copolymer resin obtained by copolymerizing ethylene and 4-methylpentene-1.

Description

It occurs as translucent powder or granules, and is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2870 cm^{-1} , 1460 cm^{-1} , 1384 cm^{-1} , 1366 cm^{-1} , 1169 cm^{-1} , 920 cm^{-1} , 730 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.85-0.94

Melting point: 120-130°C

Purity

- (1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

- (2) Heavy metals: Not more than 20 ppm (Method 2)

- (3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Chemical Pulp

Definition

Chemical Pulp is obtained by the chemical treatment of fiber to separate the fibers.

Description

It is white in color, practically odorless, and contains no foreign matter.

Purity

- (1) Lignin

Dissolve 0.1 g of phloroglucin in 15 mL of hydrochloric acid, add water to make 20 mL and drop onto this substance: no marked pink or red color develops.

- (2) Coloring matter

Immerse 10 g of this substance in 100 mL of freshly boiled and cooled water, stir and filter. Transfer 50 mL of the filtrate into a Nessler tube and observe downward: the filtrate is almost colorless.

- (3) Acidity or alkalinity

Transfer 10 mL of the filtrate (2) into a test tube 15 mm in inside diameter and add 2 drops of phenolphthalein TS: no red color develops. Separately, to 10 mL of the same solution, add 1 drop of methyl orange TS: no red color develops.

- (4) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows neither

marked fluorescence nor fluorescence by contamination.

Total ash: Not more than 0.65% (5.0 g)

Active Carbon

Definition

Active Carbon is obtained by activating carbon substance such as plant-based fibers treated with oxidizing gas or chemicals at high temperature.

Description

It occurs as black powder, granules or fibrous substances, and it is odorless.

Identification

- (1) If this substance is a powder, use it as it is. If it is in a granular or fibrous state, pulverize well to powder. Take about 0.1 g of this substance in a powder state, add 10 mL of dilute methylene blue TS and 2 drops of hydrochloric acid (1 in 4), shake well and filter with a dry filter paper for quantitative analysis (5C): the filtrate is colorless.
- (2) Take about 0.5 g of this substance in a powder state in a test tube and heat over a direct flame while supplying air: it burns without any flame. Pass the evolved gas through calcium hydroxide TS: a white turbidity is produced.

Purity

- (1) Color and acidity or alkalinity of solution
To 3.0 g of this substance, add 60 mL of water, boil for 5 minutes, cool, add water to make 60 mL and filter: the filtrate is colorless and neutral.
- (2) Heavy metals: Not more than 50 ppm (0.50 g, Method 2, Standard Lead Solution 2.5 mL)
- (3) Arsenic: Not more than 2 ppm (Method 2)

Flocculent Sodium Carboxymethylcellulose

Definition

Flocculent Sodium Carboxymethylcellulose is flocculent fiber made from the sodium salt of carboxymethylether of parts of fiber structure of plant-based fibers.

Description

- (1) It is white in color, odorless, and contains no foreign matter.
- (2) It does not remarkably contain broken pieces of pericarp and seed, or nep.

Identification

It becomes slightly viscous when water is added.

Purity

- (1) Coloring matter
Immerse 10 g of this substance in 100 mL of ethanol, press out, transfer 50 mL of the extract into a Nessler tube and observe downward: a yellow color may develop but neither blue nor green color develops.
- (2) Acidity or alkalinity

To 10 g of this substance, add 100 mL of freshly boiled and cooled water and allow to cool. To 25 mL of the solution, add 3 drops of phenolphthalein TS: no red color develops. Separately, to 25 mL of the same solution, add 1 drop of methyl orange TS: no red color develops.

(3) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows neither marked fluorescence nor fluorescence by contamination.

(4) Sedimentation velocity

Prepare a test basket, weighing about 3 g from copper wire 0.4 mm in diameter (No. 26) in the form of a cylinder 50 mm in diameter and 80 mm in depth, with 20-mm intervals between the wires. Place 5.0 g of this substance in the test basket, drop the basket on its side gently into the water about 200 mm in depth at ordinary temperature from the height of about 10 mm above the water surface: the basket sinks in water within 8 seconds.

Total ash: Not more than 5.6% (5.0 g)

Absorbent Paper

Definition

Absorbent Paper is a paper made of chemical pulp.

Description

- (1) It is white in color, odorless, and contains no foreign matter.
- (2) It does not remarkably contain undissociated fibers.

Purity

(1) Lignin

Dissolve 0.1 g of phloroglucin in 15 mL of hydrochloric acid, add water to make 20 mL and drop onto this substance: no marked pink or red color develops.

(2) Coloring matter

Immerse 10 g of this substance in 100 mL of ethanol, press out, transfer 50 mL of the extract into a Nessler tube and observe downward: a yellow color may develop but neither blue nor green color develops.

(3) Acidity or alkalinity

To 10 g of this substance, add 100 mL of freshly boiled and cooled water and allow to cool. To 25 mL of the solution, add 3 drops of phenolphthalein TS: no red color develops. Separately, to 25 mL of the same solution, add 1 drop of methyl orange TS: no red color develops.

(4) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows neither marked fluorescence nor fluorescence by contamination.

(5) Sedimentation velocity

Prepare a test basket, weighing about 3 g from copper wire 0.4 mm in diameter (No. 26) in the form of a cylinder 50 mm in diameter and 80 mm in depth, with 20-mm intervals between the wires. Place 5.0 g of

this substance in the test basket, drop the basket on its side gently into the water about 200 mm in depth at ordinary temperature from the height of about 10 mm above the water surface: the basket sinks in water within 8 seconds.

Total ash: Not more than 0.65% (5.0 g)

High-density Polyethylene (HDPE)

Definition

High-density Polyethylene (HDPE) is a straight-chain high-density polyethylene resin obtained by the polymerization of ethylene.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2870 cm^{-1} , 1460 cm^{-1} , 730 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.85-1.00

Melting point: 115-140°C

Purity

(1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

(2) Heavy metals: Not more than 20 ppm (Method 2)

(3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Cycloaliphatic Saturated Hydrocarbon Resin

Definition

Cycloaliphatic Saturated Hydrocarbon Resin is a hydrogenated C_9 -group petroleum resin. The mean molecular weight is 550 to 900.

Description

It occurs as an almost colorless clear glass mass, and it is odorless or has a faint, characteristic odor.

Specific gravity: 0.98-1.03

Identification

Dissolve about 1.0 g of this substance in 5 mL of chloroform and determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2930 cm^{-1} , 1450 cm^{-1} , 1380 cm^{-1} and 760 cm^{-1} .

Heavy metals: Not more than 10 ppm (Method 2)

Loss on drying: Not more than 1.0% (2.0 g, 105°C, 2 hours)

Residue on ignition: Not more than 0.01% (50 g, 800°C, 3 hours)

Cycloparaffin

Definition

Cycloparaffin is a mixture of liquid hydrocarbons obtained from petroleum.

Description

It is a colorless to light yellow liquid, and it is odorless or has a faint, characteristic odor.

Specific gravity: d_4^{15} 0.81-0.94

Purity

(1) Acidity or alkalinity

To 10 mL of this substance, add 10 mL of hot water and 1 drop of phenolphthalein TS, and shake vigorously: no red color develops. Separately, to 10 mL of this substance, add 10 mL of hot water and 1 drop of methyl orange TS: no red color develops.

(2) Sulfur compounds

To 4 mL of this substance, add 2 mL of ethanol (99.5) and 2 drops of a transparent sodium hydroxide solution (1 in 5) saturated with lead monoxide, heat at 70°C for 10 minutes with occasional shaking, and allow to cool: no dark brown color develops.

(3) Polynuclear aromatic hydrocarbons

Transfer 25 mL of this substance into a 100-mL separator using a 25-mL measuring cylinder, wash the measuring cylinder with 25 mL of n-hexane for ultraviolet-visible spectrophotometry, combine the washings with the liquid in the separator, and shake well. Shake this solution vigorously with 5.0 mL of dimethylsulfoxide for ultraviolet-visible spectrophotometry for 2 minutes, and allow to stand for 15 minutes. Transfer the lower layer into a 50-mL separator, add 2 mL of n-hexane for ultraviolet-visible spectrophotometry, shake vigorously for 2 minutes and allow to stand for 2 minutes. Transfer the lower layer into a glass-stoppered 10-mL centrifuge tube, centrifuge at the rate between 2500 and 3000 rpm for about 10 minutes. Transfer the clear solution so obtained into a cell, stopper tightly, and use this solution as the sample solution. Separately, transfer 25 mL of n-hexane for ultraviolet-visible spectrophotometry into another 50-mL separator, shake vigorously with 5.0 mL of dimethylsulfoxide for ultraviolet-visible spectrophotometry for 2 minutes, and allow to stand for 2 minutes. Transfer the lower layer into a glass-stoppered 10-mL centrifuge tube, centrifuge at the rate between 2500 and 3000 rpm for about 10 minutes. Transfer the clear solution so obtained into a cell, and stopper tightly. Immediately determine the absorbance of the sample solution using this solution as the blank: it is not more than 4.0 at the wavelength between 260 and 350 nm.

(4) Heavy metals: Not more than 10 ppm (2.0 g, Method 3, Standard Lead Solution 2.0 mL)

(5) Arsenic: Not more than 2 ppm (Method 2)

Dibenzothiazyl Disulfide

Definition

Dibenzothiazyl Disulfide is di(benzothiazolyl-2)disulfide ($C_{14}H_8N_2S_4$ 332.49) obtained by the reaction of an

aqueous solution of the alkali salt of 2-mercaptobenzothiazole with oxidants such as bromine.

Description

It occurs as a white to light yellow powder, and it is not soluble in water and ethanol, but soluble in toluene and chloroform.

Identification

Take 0.1 g of this substance in a volumetric flask and dissolve in chloroform to make 100 mL. Perform the test with 5 μL of this solution as directed under Liquid Chromatography: a peak is observed at the retention time of about 22.6 minutes.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 254 nm)

Column: A stainless steel column, 4.6 mm in inside diameter and 25 cm in length, packed with octadecylsilanized silica gel

Flow rate: 0.8 mL/min

Melting point: 165-175°C

Purity

(1) Heavy metals: Not more than 20 ppm (Method 2)

(2) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.5% (Method 1)

Aliphatic Hydrocarbon Resin

Definition

Aliphatic Hydrocarbon Resin is a C_5 -group petroleum hydrocarbon resin. The mean molecular weight is 500 to 2000.

Description

(1) It occurs as a white to light yellowish brown easily-breakable solid, and it is odorless or has a faint, characteristic odor.

(2) It is freely soluble in toluene, and practically insoluble in water and ethanol.

Specific gravity: 1.03-1.06

Identification

Mix this substance, previously pulverized, with potassium bromide powder, solidify to plate-like shape, and determine the infrared absorption spectrum of this substance as directed in the potassium bromide disk method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about between 2970 cm^{-1} and 2950 cm^{-1} and 1300 cm^{-1} .

Heavy metals: Not more than 10 ppm (Method 2)

Loss on drying: Not more than 1.0% (2.0 g, 105°C, 2 hours)

Residue on ignition: Not more than 0.01% (1.0 g, 450-550°C)

Aliphatic and Aromatic Copolymer Resin

(Aromatic and Denatured Aliphatic Hydrocarbon Resin)

Definition

Aliphatic and Aromatic Copolymer Resin (Aromatic and Denatured Aliphatic Hydrocarbon Resin) is obtained by the copolymerization of a C₅-group resin and an aromatic resin. The mean molecular weight is 400 to 1500.

Description

- (1) It occurs as a light yellow solid or viscous liquid, and it is practically odorless.
- (2) It is practically insoluble in water and ethanol, but freely soluble in tetrahydrofuran and diethyl ether.

Identification

Dissolve 1 g of this substance in 10 mL of toluene, apply onto a potassium bromide disk, evaporate the toluene to obtain a film and determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about between 2970 cm⁻¹ and 2950 cm⁻¹, 1600 cm⁻¹, 1460 cm⁻¹, 1370 cm⁻¹ and 700 cm⁻¹.

Purity

- (1) Clarity of solution

Dissolve 150 g of this substance in 400 mL of tetrahydrofuran: the solution is clear.

- (2) Heavy metals: Not more than 10 ppm (Method 2)

Loss on drying: Not more than 3% (5.0 g, 105°C, 4 hours)

Residue on ignition: Not more than 0.01% (30 g, 600°C)

Aliphatic Saturated Hydrocarbon Resin

Definition

Aliphatic Saturated Hydrocarbon Resin is a hydrogenated C₅-group petroleum resin. The mean molecular weight is 300 to 600.

Description

It is an almost colorless clear viscous liquid, and it is odorless or has a faint, characteristic odor.

Specific gravity: d₄¹⁵ 0.90-0.95

Identification

Dissolve about 1 g of this substance in 5 mL of chloroform and determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2930 cm⁻¹, 1450 cm⁻¹, 1380 cm⁻¹ and 760 cm⁻¹.

Heavy metals: Not more than 10 ppm (Method 2)

Loss on drying: Not more than 1.0% (2.0 g, 105°C, 2 hours)

Residue on ignition: Not more than 0.1% (50 g, 800°C, 3 hours)

Hydrogenated Aliphatic and Aromatic Copolymer Resin

Definition

Hydrogenated Aliphatic and Aromatic Copolymer Resin is obtained by the copolymerization and hydrogenation of a C₅-group resin and an aromatic resin. The mean molecular weight is 500 to 1000.

Description

It occurs as a colorless clear pellet or flake solid, and it is odorless or has a faint, characteristic odor. It is freely soluble in toluene, xylene and diethyl ether, but practically insoluble in water and ethanol.

Identification

Mix completely 1 mg of this substance and 100 to 200 mg of dried potassium bromide for infrared spectrophotometry, and determine the infrared absorption spectrum of this substance as directed in the potassium bromide disk method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 3650 cm^{-1} , 2930 cm^{-1} , 2850 cm^{-1} , 2790 cm^{-1} , 2670 cm^{-1} , 2600 cm^{-1} , 1746 cm^{-1} , 1449 cm^{-1} , 1375 cm^{-1} , 890 cm^{-1} , 843 cm^{-1} , 757 cm^{-1} and 700 cm^{-1} .

Heavy metals: Not more than 10 ppm (Method 2)

Loss on drying: Not more than 1.0% (2.0 g, 105°C , 2 hours)

Residue on ignition: Not more than 0.02% (5.0 g, 850°C , 30 minutes)

Hydrogenated Dicyclopentadiene-group Hydrocarbon Resin

Definition

Hydrogenated Dicyclopentadiene-group Hydrocarbon Resin is a solid resin obtained by the hydrogenation of a dicyclopentadiene-based polymer. The mean molecular weight is 300 to 700.

Description

It occurs as a colorless, clear, easily-breakable solid, and it is odorless.

It is freely soluble in tetrahydrofuran and toluene, but practically insoluble in water and ethanol.

Acid value: Not more than 0.1 (Method 1)

Weigh accurately 2 g of this substance and dissolve it in a 40-mL mixture of toluene and isopropyl alcohol (2:1), and perform the test with this solution.

Specific gravity: 1.05-1.08

Identification

Mix this substance, previously pulverized, with potassium bromide powder, solidify to plate-like shape, and determine the infrared absorption spectrum of this substance as directed in the potassium bromide disk method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about between 2970 cm^{-1} and 2950 cm^{-1} , 1463 cm^{-1} and 1373 cm^{-1} .

Purity

(1) Clarity of solution

Dissolve 150 g of this substance in 400 mL of tetrahydrofuran: the solution is clear.

(2) Heavy metals: Not more than 10 ppm (Method 2)

Loss on drying: Not more than 1% (5.0 g, 105°C , 4 hours)

Residue on ignition: Not more than 0.01% (30 g, 600°C)

Styrene-Isoprene-Styrene Block Copolymer

Definition

Styrene-Isoprene-Styrene Block Copolymer is a ternary block copolymer of polystyrene, polyisoprene and polystyrene. The mean molecular weight is 80000 to 200000.

Description

It occurs as a white to light yellow elastic pellet, crumb or powder solid, and it is odorless or has a faint, characteristic odor.

It is freely soluble in tetrahydrofuran, diethyl ether, and toluene, but practically insoluble in water and ethanol.

Identification

Dissolve 1 g of this substance in 10 mL of toluene, apply 1 drop of this solution onto a potassium bromide disk, evaporate the solvent to obtain a film and determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2850 cm^{-1} , 1600 cm^{-1} , 1452 cm^{-1} , 1375 cm^{-1} , and 837 cm^{-1} .

Viscosity

Dissolve 50.0 g of this substance in 150 g of toluene, use the solution as the sample solution, and measure viscosity twice: the mean viscosity is 100 - 1700 mPa·s. (Brookfield type viscometer, No. 3, 10-60 rotations, $30\pm 1^\circ\text{C}$, 1 minute)

Purity

(1) Clarity and color of solution

Dissolve 1.0 g of this substance in 100 mL of toluene: the solution is colorless and clear.

(2) Extractable substances

To 5.0 g of this substance, add 80 mL of water, and boil under a reflux condenser for 30 minutes. After cooling, filter the extract, and add water to the filtrate to make exactly 100 mL. Use this solution as the sample solution for the following tests.

1) pH: 6.0-9.0

2) Chloride

Perform the test with 10 mL of the sample solution as directed under Chloride Limit Test: not more than 0.085%. Prepare the control solution with 1.2 mL of 0.01 mol/L hydrochloric acid

3) Heavy metals: Not more than 20 ppm (20 mL of the sample solution, Method 1, Standard Lead Solution 2 mL)

4) Potassium permanganate-reducing substances

Transfer 25 mL of the sample solution into a glass-stoppered, Erlenmyer flask, add 10.0 mL of 0.002 mol/L potassium permanganate VS and 5 mL of dilute sulfuric acid, and boil for 3 minutes. After cooling, add 0.10 g of potassium iodide, stopper tightly, shake, and allow to stand for 10 minutes. Titrate the solution with 0.01 mol/L sodium thiosulfate VS (indicator: 5 drops of starch TS). Perform the test in the same manner, using 25 mL of the blank solution, and obtain the difference of the consumption of 0.002 mol/L potassium permanganate VS between these solutions: not more than 2.0 mL.

(3) Styrene

Weigh exactly 5.0 g of this substance, and dissolve in 50 mL of tetrahydrofuran. Add methanol to make

exactly 100 mL, shake vigorously for 10 minutes, centrifuge and use the supernatant liquid as the sample solution. Separately, weigh exactly 0.10 g of styrene, and add methanol to make exactly 100 mL. Measure exactly 5 mL of the solution, and add methanol to make exactly 100 mL. Measure exactly 1 mL of the solution, add 50 mL of tetrahydrofuran, mix well, add methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 100 μ L each of the sample solution and standard solution as directed under Liquid Chromatography according to the following conditions. Determine each peak area from these solutions by the automatic integration method: the peak area of styrene from the sample solution is not larger than the peak area of styrene from the standard solution.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 268 nm)

Column: A stainless steel column, about 4 mm in inside diameter and about 15 cm in length, packed with 10- μ m octadecylsilanized silica gel for liquid chromatography

Column temperature: A constant temperature of about 25°C

Mobile phase: A mixture of water and tetrahydrofuran (1:1)

Flow rate: Adjust the flow rate so that the retention time of styrene is about 5 minutes.

Detection sensitivity: Adjust it so that the peak height of styrene obtained from 100 μ L of the standard solution is not less than 5 mm.

(4) Lithium

Take 1.0 g of this substance in a crucible, and ignite at 450°C to 500°C to incinerate. After cooling, dissolve the residue in 2 mL of 0.1 mol/L hydrochloric acid TS, add 10 mL of water, and filter through a glass filter (G4). To this filtrate, add water to make exactly 200 mL, and use this solution as the sample solution. Separately, measure exactly 1.0 mL of Standard Lithium Solution for Atomic Absorption Spectrophotometry, and add water to make exactly 100 mL. Measure exactly 10 mL of this solution, add 2 mL of 0.1 mol/L hydrochloric acid TS, add water to make exactly 100 mL, and use this solution as the standard solution. Perform the test with the sample solution and the standard solution as directed under Atomic Absorption Spectrophotometry according to the following conditions: the absorbance of the sample solution is not more than that of the standard solution.

Gas: Combustible gas-Acetylene

Supporting gas-Air

Lamp: Lithium hollow-cathode lamp

Wavelength: 670.8nm

Loss on drying: Not more than 1.0% (0.1 g, 105°C, 4 hours)

Residue on ignition: Not more than 2.0% (1 g)

Styrene-Ethylene-Butylene-Styrene Block Copolymer

Definition

Styrene-Ethylene-Butylene-Styrene Block Copolymer is a block copolymer of polystyrene-polyethylene butylene-polystyrene, obtained by the hydrogenation of a copolymer consisting of a

polystyrene-polybutadiene-polystyrene block. The mean molecular weight is 30000 to 300000.

Description

It occurs as a white to light yellow elastic pellet, crumb or powder solid, and it is odorless or has a faint, characteristic odor.

It is freely soluble in tetrahydrofuran, diethyl ether, and toluene, but practically insoluble in water and ethanol.

Identification

Dissolve 1 g of this substance in 10 mL of toluene, apply 1 drop of this solution onto a potassium bromide disk, evaporate the solvent to obtain a film and determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2920 cm^{-1} , 2850 cm^{-1} , 1601 cm^{-1} , 1380 cm^{-1} , 760 cm^{-1} and 700 cm^{-1} .

Viscosity

Dissolve 50.0 g of this substance in 150 g of toluene, remove air bubbles, use the solution as the sample solution, and measure viscosity twice: the mean viscosity is 100 - 10000 mPa·s. (Brookfield type viscometer, No. 3, 10-60 rotations, $25\pm 1^\circ\text{C}$, 1 minute)

Purity

(1) Clarity and color of solution

Dissolve 1.0 g of this substance in 100 mL of toluene: the solution is colorless and clear.

(2) Extractable substances

To 5.0 g of this substance, add 80 mL of water, and boil under a reflux condenser for 30 minutes. After cooling, filter the extract, and add water to the filtrate to make exactly 100 mL. Use this solution as the sample solution for the following tests.

1) pH: 5.0-9.0

2) Chloride

Perform the test with 10 mL of the sample solution as directed under Chloride Limit Test: not more than 0.085%. Prepare the control solution with 1.2 mL of 0.01 mol/L hydrochloric acid

3) Heavy metals: Not more than 20 ppm (20 mL of the sample solution, Method 1, Standard Lead Solution 2 mL)

4) Potassium permanganate-reducing substances

Transfer 25 mL of the sample solution into a glass-stoppered, Erlenmyer flask, add 10.0 mL of 0.002 mol/L potassium permanganate VS and 5 mL of dilute sulfuric acid, and boil for 3 minutes. After cooling, add 0.10 g of potassium iodide, stopper tightly, shake and allow to stand for 10 minutes. Titrate the solution with 0.01 mol/L sodium thiosulfate VS (indicator: 5 drops of starch TS). Perform the test in the same manner, using 25 mL of the blank solution, and obtain the difference of the consumption of 0.002 mol/L potassium permanganate VS between these solutions: not more than 2.0 mL.

(3) Styrene

Weigh exactly 5.0 g of this substance, and dissolve in 50 mL of tetrahydrofuran. Add methanol to make exactly 100 mL, shake vigorously for 10 minutes, centrifuge and use the supernatant liquid as the sample

solution. Separately, weigh exactly 0.10 g of styrene, and add methanol to make exactly 100 mL. Measure exactly 5 mL of the solution, and add methanol to make exactly 100 mL. Measure exactly 1 mL of the solution, add 50 mL of tetrahydrofuran, mix well, add methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 100 μ L each of the sample solution and standard solution as directed under Liquid Chromatography according to the following conditions. Determine each peak area from these solutions by the automatic integration method: the peak area of styrene from the sample solution is not larger than the peak area of styrene from the standard solution.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 268 nm)

Column: A stainless steel column, about 4 mm in inside diameter and about 15 cm in length, packed with 10- μ m octadecylsilanized silica gel for liquid chromatography

Column temperature: A constant temperature of about 25°C

Mobile phase: A mixture of water and tetrahydrofuran (1:1)

Flow rate: Adjust the flow rate so that the retention time of styrene is about 5 minutes.

Detection sensitivity: Adjust it so that the peak height of styrene obtained from 100 μ L of the standard solution is not less than 5 mm.

(4) Lithium

Take 1.0 g of this substance in a crucible, and ignite at 450°C to 500°C to incinerate. After cooling, dissolve it in 2 mL of 0.1 mol/L hydrochloric acid TS, add 10 mL of water, and filter through a glass filter (G4). To this filtrate add water to make exactly 200 mL, and use this solution as the sample solution. Separately, measure exactly 1.0 mL of Standard Lithium Solution for Atomic Absorption Spectrophotometry, and add water to make exactly 100 mL. Measure exactly 10 mL of this solution, add 2 mL of 0.1 mol/L hydrochloric acid TS, add water to make exactly 100 mL, and use this solution as the standard solution. Perform the test with the sample solution and the standard solution as directed under Atomic Absorption Spectrophotometry according to the following conditions: the absorbance of the sample solution is not more than that of the standard solution.

Gas: Combustible gas-Acetylene

Supporting gas-Air

Lamp: Lithium hollow-cathode lamp

Wavelength: 670.8nm

Loss on drying: Not more than 1.0% (1.0 g, 105°C, 4 hours)

Residue on ignition: Not more than 2.0% (Method 1)

Styrene-Ethylene-Propylene-Styrene Block Copolymer

Definition

Styrene-Ethylene-Propylene-Styrene Block Copolymer is a block copolymer of polystyrene-polyethylene propylene-polystyrene, obtained by the hydrogenation of a copolymer consisting of a polystyrene-polyisoprene-polystyrene block or styrene-poly(isoprene/butadiene)-polystyrene. The mean

molecular weight is 30000 to 300000.

Description

It occurs as a white to light yellow elastic pellet, crumb or powder solid, and it is odorless or has a faint, characteristic odor.

It is freely soluble in tetrahydrofuran, diethyl ether, and toluene, but practically insoluble in water and ethanol.

Identification

Dissolve 1 g of this substance in 10 mL of toluene, apply 1 drop of this solution onto a potassium bromide disk, evaporate the solvent to obtain a film and determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2920 cm^{-1} , 2850 cm^{-1} , 1601 cm^{-1} , 1380 cm^{-1} , 760 cm^{-1} and 700 cm^{-1} .

Viscosity

Dissolve 50.0 g of this substance in 150 g of toluene, remove air bubbles, use the solution as the sample solution, and measure viscosity twice: the mean viscosity is 150 - 20000 mPa·s. (Brookfield type viscometer, No. 3, 10-60 rotations, $25\pm 1^\circ\text{C}$, 1 minute)

Purity

(1) Clarity and color of solution

Dissolve 1.0 g of this substance in 100 mL of toluene: the solution is colorless and clear.

(2) Extractable substances

To 5.0 g of this substance, add 80 mL of water, and boil under a reflux condenser for 30 minutes. After cooling, filter the extract, and add water to the filtrate to make exactly 100 mL. Use this solution as the sample solution for the following tests.

1) pH: 5.0-9.0

2) Chloride

Perform the test with 10 mL of the sample solution as directed under Chloride Limit Test: not more than 0.085%. Prepare the control solution with 1.2 mL of 0.01 mol/L hydrochloric acid

3) Heavy metals: Not more than 20 ppm (20 mL of the sample solution, Method 1, Standard Lead Solution 2 mL)

4) Potassium permanganate-reducing substances

Transfer 25 mL of the sample solution into a glass-stoppered, Erlenmyer flask, add 10.0 mL of 0.002 mol/L potassium permanganate VS and 5 mL of dilute sulfuric acid, and boil for 3 minutes. After cooling, add 0.10 g of potassium iodide, stopper tightly, shake and allow to stand for 10 minutes. Titrate the solution with 0.01 mol/L sodium thiosulfate VS (indicator: 5 drops of starch TS). Perform the test in the same manner, using 25 mL of the blank solution, and obtain the difference of the consumption of 0.002 mol/L potassium permanganate VS between these solutions: not more than 2.0 mL.

(3) Styrene

Weigh exactly 5.0 g of this substance, and dissolve in 50 mL of tetrahydrofuran. Add methanol to make exactly 100 mL, shake vigorously for 10 minutes, centrifuge and use the supernatant liquid as the sample

solution. Separately, weigh exactly 0.10 g of styrene, and add methanol to make exactly 100 mL. Measure exactly 5 mL of the solution, and add methanol to make exactly 100 mL. Measure exactly 1 mL of the solution, add 50 mL of tetrahydrofuran, mix well, add methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 100 μ L each of the sample solution and standard solution as directed under Liquid Chromatography according to the following conditions. Determine each peak area from these solutions by the automatic integration method: the peak area of styrene from the sample solution is not larger than the peak area of styrene from the standard solution.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 268 nm)

Column: A stainless steel column, about 4 mm in inside diameter and about 15 cm in length, packed with 10- μ m octadecylsilanized silica gel for liquid chromatography

Column temperature: A constant temperature of about 25°C

Mobile phase: A mixture of water and tetrahydrofuran (1:1)

Flow rate: Adjust the flow rate so that the retention time of styrene is about 5 minutes.

Detection sensitivity: Adjust it so that the peak height of styrene obtained from 100 μ L of the standard solution is not less than 5 mm.

(4) Lithium

Take 1.0 g of this substance in a crucible, and ignite at 450°C to 500°C to incinerate. After cooling, dissolve it in 2 mL of 0.1 mol/L hydrochloric acid TS, add 10 mL of water, and filter through a glass filter (G4). To this filtrate add water to make exactly 200 mL, and use this solution as the sample solution.

Separately, measure exactly 1.0 mL of Standard Lithium Solution for Atomic Absorption

Spectrophotometry, and add water to make exactly 100 mL. Measure exactly 10 mL of this solution, add 2 mL of 0.1 mol/L hydrochloric acid TS, add water to make exactly 100 mL, and use this solution as the standard solution. Perform the test with the sample solution and the standard solution as directed under Atomic Absorption Spectrophotometry according to the following conditions: the absorbance of the sample solution is not more than that of the standard solution.

Gas: Combustible gas-Acetylene

Supporting gas-Air

Lamp: Lithium hollow-cathode lamp

Wavelength: 670.8nm

Loss on drying: Not more than 1.0% (1.0 g, 105°C, 4 hours)

Residue on ignition: Not more than 2.0% (Method 1)

Styrene-Butadiene-Styrene Block Copolymer

Definition

Styrene-Butadiene-Styrene Block Copolymer is a block copolymer of polystyrene and polybutadiene. The mean molecular weight is 30000 to 300000.

Description

It occurs as a white to light yellow elastic pellet, crumb or powder solid, and it is odorless or has a faint, characteristic odor.

It is freely soluble in tetrahydrofuran and toluene, but practically insoluble in water and ethanol.

Identification

Dissolve 1 g of this substance in 10 mL of toluene, apply 1 drop of this solution onto a potassium bromide disk, evaporate the solvent to obtain a film and determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2850 cm^{-1} , 1600 cm^{-1} , 1452 cm^{-1} , 965 cm^{-1} , 910 cm^{-1} , and 700 cm^{-1} .

Viscosity

Dissolve 50.0 g of this substance in 150 g of toluene, use the solution as the sample solution, and measure viscosity twice: the mean viscosity is 200 - 20000 mPa·s. (Brookfield type viscometer, No. 3, 10-60 rotations, $25\pm 1^\circ\text{C}$, 1 minute)

Purity

(1) Clarity and color of solution

Dissolve 1.0 g of this substance in 100 mL of toluene: the solution is colorless and clear.

(2) Extractable substances

To 5.0 g of this substance, add 80 mL of water, and boil under a reflux condenser for 30 minutes. After cooling, filter the extract, and add water to the filtrate to make exactly 100 mL. Use this solution as the sample solution for the following tests.

1) pH: 5.0-9.0

2) Chloride

Perform the test with 10 mL of the sample solution as directed under Chloride Limit Test: not more than 0.085%. Prepare the control solution with 1.2 mL of 0.01 mol/L hydrochloric acid

3) Heavy metals: Not more than 20 ppm (20 mL of the sample solution, Method 1, Standard Lead Solution 2 mL)

4) Potassium permanganate-reducing substances

Transfer 25 mL of the sample solution into a glass-stoppered, Erlenmyer flask, add 10.0 mL of 0.002 mol/L potassium permanganate VS and 5 mL of dilute sulfuric acid, and boil for 3 minutes.

After cooling, add 0.10 g of potassium iodide, stopper tightly, shake and allow to stand for 10 minutes.

Titrate the solution with 0.01 mol/L sodium thiosulfate VS (indicator: 5 drops of starch TS). Perform the test in the same manner, using 25 mL of the blank solution, and obtain the difference of the consumption of 0.002 mol/L potassium permanganate VS between these solutions: not more than 2.0 mL.

(3) Styrene

Weigh exactly 5.0 g of this substance, and dissolve in 50 mL of tetrahydrofuran. Add methanol to make exactly 100 mL, shake vigorously for 10 minutes, centrifuge and use the supernatant liquid as the sample solution. Separately, weigh exactly 0.10 g of styrene, and add methanol to make exactly 100 mL. Measure exactly 5 mL of the solution, and add methanol to make exactly 100 mL. Measure exactly 1 mL of the

solution, add 50 mL of tetrahydrofuran, mix well, add methanol to make exactly 100 mL, and use this solution as the standard solution. Perform the test with 100 μ L each of the sample solution and standard solution as directed under Liquid Chromatography according to the following conditions. Determine each peak area from these solutions by the automatic integration method: the peak area of styrene from the sample solution is not larger than the peak area of styrene from the standard solution.

Operating conditions

Detector: An ultraviolet absorption photometer (wavelength: 268 nm)

Column: A stainless steel column, about 4 mm in inside diameter and about 15 cm in length, packed with 10- μ m octadecylsilanized silica gel for liquid chromatography

Column temperature: A constant temperature of about 25°C

Mobile phase: A mixture of water and tetrahydrofuran (1:1)

Flow rate: Adjust the flow rate so that the retention time of styrene is about 5 minutes.

Detection sensitivity: Adjust it so that the peak height of styrene obtained from 100 μ L of the standard solution is not less than 5 mm.

(4) Lithium

Take 1.0 g of this substance in a crucible, and ignite at 450°C to 500°C to incinerate. After cooling, dissolve it in 2 mL of 0.1 mol/L hydrochloric acid TS, add 10 mL of water, and filter through a glass filter (G4). To this filtrate add water to make exactly 200 mL, and use this solution as the sample solution.

Separately, measure exactly 1.0 mL of Standard Lithium Solution for Atomic Absorption

Spectrophotometry, and add water to make exactly 100 mL. Measure exactly 10 mL of this solution, add 2 mL of 0.1 mol/L hydrochloric acid TS, add water to make exactly 100 mL, and use this solution as the standard solution. Perform the test with the sample solution and the standard solution as directed under Atomic Absorption Spectrophotometry according to the following conditions: the absorbance of the sample solution is not more than that of the standard solution.

Gas: Combustible gas-Acetylene

Supporting gas-Air

Lamp: Lithium hollow-cathode lamp

Wavelength: 670.8nm

Loss on drying: Not more than 1.0% (1.0 g, 105°C, 4 hours)

Residue on ignition: Not more than 2.0% (Method 1)

Styrene-Methacrylate Copolymer Solution

Definition

Styrene-Methacrylate Copolymer Solution is an aqueous solution obtained by quaternizing copolymers of styrene and methacrylic acid esters with epichlorohydrin.

Description

It is a white to milky white liquid, and it has a faint odor of acetic acid.

Identification

Determine the infrared absorption spectrum of this substance, previously dried at 105°C for about 2 hours, as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2920 cm⁻¹, 1730 cm⁻¹, 1490 cm⁻¹, 1450 cm⁻¹, 1380 cm⁻¹, 760 cm⁻¹ and 700 cm⁻¹.

pH: 4.0 - 6.0 (1 in 10)

Purity

- (1) Heavy metals: Not more than 10 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Method 2)
- (3) Epichlorohydrin

To 50 g of this substance, add 200 mL of water, and extract the solution with 30 mL of diethyl ether 5 times. Combine the ether extracts, wash with 30 mL of water, dehydrate with 5 g of anhydrous sodium sulfate, and evaporate the ether. Dissolve the residue in 5 mL of acetone, and use this solution as the sample solution. Separately, take 5 mL of acetone solution of epichlorohydrin (1 in 10000) and use this solution as the standard solution. Perform the test with each of the sample solution and standard solution as directed under Gas Chromatography according to the following conditions: the peak area of epichlorohydrin from the sample solution is not larger than the peak area of epichlorohydrin from the standard solution.

Operating conditions

Detector: Hydrogen flame-ionization detector

Column: A column 3-4 mm in inside diameter packed with siliceous earth for gas chromatography (177 to 250 μm) coated with polyethylene glycol 20 M in 10%

Column temperature: From 80 to 140°C

Heating rate: 10°C/min

Carrier gas and flow rate: Adjust the flow rate so that the retention time of nitrogen and epichlorohydrin is about 4 minutes.

Injection volume of sample: 10 μL

Hydrophobic Zeolite

Definition

Hydrophobic Zeolite is hydrophobic zeolite obtained by the reaction of sodium silicate and sodium aluminate.

Description

It is white in color, practically odorless, and contains no foreign matter.

Identification

- (1) To 0.1 g of this substance, add 1 mL of 1 mol/L hydrochloric acid, disperse by ultrasonication for 30 seconds, and boil for 5 minutes. After cooling, add 2 mL of water, and filter the solution through a membrane filter with a pore size of 0.45 μm. Add ammonia TS to the filtrate until a white, gelatinous precipitate is produced. Add 5 drops of alizarin S TS: the precipitate changes to red.
- (2) Prepare a bead by fusing ammonium sodium hydrogenphosphate tetrahydrate on a platinum loop. Place the bead in contact with this substance and fuse again: an infusible matter appears in the bead, which

changes to an opaque bead with a web-like structure upon cooling.

Purity

(1) Acidity or alkalinity

To 5.0 g of this substance, add 70 mL of water, shake vigorously and boil for 5 minutes. After cooling, add water to make 100 mL, shake well, and centrifuge: the supernatant liquid is neutral.

(2) Heavy metals

Disperse 1.0 g of this substance in 2 mL of water, add 10 mL of dilute hydrochloric acid, shake well, and filter. Wash the residue with 10 mL of water, combine the washing with the filtrate. Add ammonia solution (28) dropwise until a precipitate just appears, and add dropwise dilute hydrochloric acid with vigorous shaking to redissolve the residue. To the solution, add 0.15 g of hydroxylamine hydrochloride, and heat. After cooling, add 0.15 g of sodium acetate, 2 mL of dilute acetic acid and water to make 50 mL. Perform the test using this solution as the sample solution as directed in Method 4: not more than 30 ppm. To 3.0 mL of Standard Lead Solution, add 0.15 g of hydroxylamine hydrochloride, 0.15 g of sodium acetate, 2 mL of dilute acetic acid and water to make 50 mL, and use this solution as the control solution.

(3) Arsenic

Disperse 0.4 g of this substance in 1 mL of water, add 10 mL of dilute hydrochloric acid, and shake well. Perform the test using this solution as the sample solution: not more than 5 ppm.

Loss on drying: Not more than 4.0% (1 g, 105°C, 2 hours)

Absorbent Cotton

Definition

Absorbent Cotton is defatted cotton.

Description

- (1) It is white in color, odorless, and contains no foreign matter.
- (2) It does not remarkably contain broken pieces of pericarp and seed, or nep.

Identification

It is soluble in ammonium copper TS.

Purity

(1) Coloring matter

Immerse 10 g of this substance in 100 mL of ethanol, press out, transfer 50 mL of the extract into a Nessler tube and observe downward: a yellow color may develop but neither blue nor green color develops.

(2) Acidity or alkalinity

Immerse 10 g of this substance in 100 mL of freshly boiled and cooled water. To 25 mL of the solution, add 3 drops of phenolphthalein TS: no red color develops. Separately, to 25 mL of the same solution, add 1 drop of methyl orange TS: no red color develops.

(3) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows neither

marked fluorescence nor fluorescence by contamination.

(4) Sedimentation velocity

Prepare a test basket, weighing about 3 g from copper wire 0.4 mm in diameter (No. 26) in the form of a cylinder 50 mm in diameter and 80 mm in depth, with 20-mm intervals between the wires. Place 5.0 g of this substance in the test basket, drop the basket on its side gently into the water about 200 mm in depth at ordinary temperature from the height of about 10 mm above the water surface: the basket sinks in water within 8 seconds.

Total ash: Not more than 0.25% (5.0 g)

Linear Low-density Polyethylene (LLDPE)

Definition

Linear Low-density Polyethylene (LLDPE) is a straight-chain low-density polyethylene resin with short-chain branches obtained by the polymerization of ethylene.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2870 cm^{-1} , 1460 cm^{-1} , 730 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.85-0.94

Melting point: 90-130°C

Purity

(1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

(2) Heavy metals: Not more than 20 ppm (Method 2)

(3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Low-density Polyethylene (LDPE)

Definition

Low-density Polyethylene (LDPE) is a branched low-density polyethylene resin with long-chain branches obtained by the polymerization of ethylene.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2870 cm^{-1} , 1460 cm^{-1} , 1384 cm^{-1} , 1379 cm^{-1} , 1366 cm^{-1} , 730 cm^{-1} and 720 cm^{-1} .

Specific gravity: 0.85-0.94

Melting point: 90-120°C

Purity

- (1) Clarity and color of solution

Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

- (2) Heavy metals: Not more than 20 ppm (Method 2)

- (3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Natural Rubber Thread

Definition

Natural Rubber Thread is obtained by vulcanization of natural rubber.

Description

It occurs as a white elastomer, is practically odorless, and contains no foreign matter.

Purity

- (1) Coloring matter

Immerse 10 g of this substance in 100 mL of freshly boiled and cooled water, stir and filter. Transfer 50 mL of the filtrate into a Nessler tube and observe downward: the filtrate is almost colorless.

- (2) Acidity or alkalinity

Transfer 10 mL of the filtrate (1) into a test tube 15 mm in inside diameter and add 2 drops of phenolphthalein TS: no red color develops. Separately, to 10 mL of the same solution, add 1 drop of methyl orange TS: no red color develops.

- (3) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows neither marked fluorescence nor fluorescence by contamination.

Elasticity

Cut this substance into a 1.5-5.0-mm piece, hold both edges of the thread 100 mm apart and apply a 75-g load: it does not break within 1 minute.

Partial Sodium Salt of Starch-Acrylic Acid Graft Polymer

Definition

Partial Sodium Salt of Starch-Acrylic Acid Graft Polymer is a water-absorbing resin consisting of slightly cross-linked partial sodium salts of starch-acrylic acid graft polymer as a principal component.

Description

- (1) It occurs as a white powder and it is practically odorless.

- (2) It swells with water but is practically insoluble in water.

- (3) Melting point: Not less than 200°C (with decomposition)

Identification

- (1) To 1.0 g of this substance, add 100 mL of water, stir and allow to stand for 10 minutes: the solution becomes gelatinous.
- (2) To 10 g of the gelatinous substance (1), add 1 mL of calcium chloride TS and shake: a white precipitate is produced.
- (3) To 10 g of the gelatinous substance (1), add 1 mL of magnesium sulfate TS and shake: a white precipitate is produced.
- (4) To 10 g of the gelatinous substance (1), add 1 mL of cobalt chloride solution (1 in 25), add 2 or 3 drops of ammonium chloride TS and shake: a light red precipitate is produced. Dry the precipitate: the color changes to purple.
- (5) To 10 g of the gelatinous substance (1), add 3 drops of iodine TS: a dark blue-purple color is produced.

Purity

- (1) Coloring matter

Immerse this substance in ethanol not less than 10 times the mass of this substance, stir for 10 minutes and filter: the filtrate is a colorless clear liquid.

- (2) Acidity or alkalinity

To 1.0 g of this substance, add 500 mL of freshly boiled and cooled water and allow to cool. To 25 mL of this solution, add 3 drops of phenolphthalein TS: no red color develops. Separately, to 25 mL of the solution, add 1 drop of methyl orange TS: a yellow color develops.

- (3) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows no marked fluorescence.

- (4) Heavy metals: Not more than 20 ppm (Method 2)

- (5) Acrylic acid

Method 1

To 5.0 g of this substance, add exactly 10 mL of methanol, shake for 4 hours, allow to stand and use the supernatant liquid as the sample solution. Separately, take 0.010 g of the acrylic acid reference standard, dissolve in methanol to make exactly 200 mL and use the solution as the standard solution. Perform the test with 5 μ L each of the sample solution and standard solution as directed under Gas Chromatography. Determine the peak heights, H_t and H_s , of acrylic acid of respective solutions: H_t is not higher than H_s .

Method 2

To 1.0 g of this substance, add 250 mL of saline, stir for 2 hours, filter, and use the resultant solution as the sample solution. Separately, take 0.20 g of acrylic acid reference standard, dissolve in saline to make exactly 100 mL. To 1 mL of the solution, add saline to make exactly 250 mL and use as the standard solution. Perform the test with 20 μ L each of the sample solution and the standard solution as directed under Liquid Chromatography. Determine the peak heights, H_t and H_s , of acrylic acid of respective solutions: H_t is not higher than H_s .

Loss on drying: Not more than 15% (2.0 g, 105°C, 3 hours)

Residue on ignition: Not more than 76% (Method 1)

Absorbency

Take 1.0 g of this substance in a nylon woven fabric (10 cm in width, 20 cm in length, 255 mesh), immerse in 1000 mL of saline for 1 hour, allow to stand for 10 minutes, remove excessive water and determine absorbency: it absorbs more than 10 times its weight.

Note) Identify acrylic acid according to Method 1 or Method 2.

Cuprammonium Rayon

Definition

Cuprammonium Rayon is a cellulose fiber obtained by recycling cellulose by cuprammonium method.

Description

It occurs as colorless to light yellow fibrous substances, and it is odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the potassium bromide disk method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about between 3450 cm^{-1} and 3250 cm^{-1} , 2900 cm^{-1} , 1650 cm^{-1} , between 1430 cm^{-1} and 1370 cm^{-1} , between 1060 cm^{-1} and 970 cm^{-1} and 890 cm^{-1} .

Specific gravity: 1.49-1.51

Melting point: 260-300°C (with decomposition)

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Method 2)

Loss on drying

Allow to stand this substance at 20°C and 65% RH for 24 hours, and perform the test with 2.0 g of this substance at 105°C for 3 hours: not more than 13%.

Residue on ignition: Not more than 2.5% (Method 2)

Paraffin

Definition

Paraffin is a mixture of solid hydrocarbons obtained by the polymerization of petroleum.

Description

It occurs as a colorless to white, more or less transparent crystalline mass, and it has a faint, characteristic odor.

Melting point: 70-110°C

Purity

- (1) Acidity or alkalinity

Melt 10 g of this substance by heating, add 10 mL of hot ethanol, shake and allow to stand: the ethanol layer is neutral.

- (2) Readily carbonizable substances

Take 5 g of this substance in a Nessler tube, melt on an oil bath at 110°C and add 5 mL of sulfuric acid

(94.5- 95.5%). Heat on an oil bath at 110°C for 30 seconds: the sulfuric acid layer has no more color than that of the following control solution.

Control solution: To 3.0 mL of Ferric (II) Chloride Colorimetric Stock Solution, add 1.5 mL of Cobalt (I) Chloride Colorimetric Stock Solution and 0.5 mL of Copper Sulfate Colorimetric Stock Solution, and shake.

(3) Sulfur compounds

To 4.0 g of this substance, add 2 mL of ethanol (99.5) and 2 drops of a transparent sodium hydroxide solution (1 in 5) saturated with lead monoxide, heat at 110°C for 10 minutes with occasional shaking, and allow to cool: no dark color is produced.

(4) Heavy metals: Not more than 30 ppm (Method 3)

(5) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.05% (5.0 g, Method 1)

Paraffin Oil

Definition

Paraffin Oil is a mixture of liquid hydrocarbons obtained from petroleum.

Description

It is a colorless, clear, oily liquid having no fluorescence, and it is odorless or has a faint odor of petroleum while hot.

Specific gravity: d_{20}^{20} 0.81-0.91

Purity

(1) Acidity or alkalinity

Boil 10 mL of this substance with 10 mL of ethanol: the ethanol layer is neutral.

(2) Sulfur compounds

To 4.0 mL of this substance, add 2 mL of ethanol (99.5) and 2 drops of a transparent sodium hydroxide solution (1 in 5) saturated with lead monoxide, heat at 70°C for 10 minutes with occasional shaking, and allow to cool: no dark color is produced.

(3) Polynuclear aromatic hydrocarbons

Transfer 25 mL of this substance into a 100-mL separator using a 25-mL measuring cylinder, wash the measuring cylinder with 25 mL of n-hexane for ultraviolet-visible spectrophotometry, combine the washings with the liquid in the separator, and shake well. Shake this solution vigorously with 5.0 mL of dimethylsulfoxide for ultraviolet-visible spectrophotometry for 2 minutes, and allow to stand for 15 minutes. Transfer the lower layer into a 50-mL separator, add 2 mL of n-hexane for ultraviolet-visible spectrophotometry, shake vigorously for 2 minutes and allow to stand for 2 minutes. Transfer the lower layer into a glass-stoppered 10-mL centrifuge tube, centrifuge at the rate between 2500 and 3000 rpm for about 10 minutes. Transfer the clear solution so obtained into a cell, stopper tightly, and use this solution as the sample solution. Separately, transfer 25 mL of n-hexane for ultraviolet-visible spectrophotometry into another 50-mL separator, shake vigorously with 5.0 mL of dimethylsulfoxide for ultraviolet-visible

spectrophotometry for 2 minutes, and allow to stand for 2 minutes. Transfer the lower layer into a glass-stoppered 10-mL centrifuge tube, centrifuge at the rate between 2500 and 3000 rpm for about 10 minutes. Transfer the clear solution so obtained into a cell, and stopper tightly. Immediately determine the absorbance of the sample solution using this solution as the blank: it is not more than 0.20 at the wavelength between 260 and 350 nm.

- (4) Heavy metals: Not more than 30 ppm (Method 3)
- (5) Arsenic: Not more than 2 ppm (Method 2)

Amorphous Propylene-Ethylene Copolymer

Definition

Amorphous Propylene-Ethylene Copolymer is a copolymer of propylene and ethylene. The mean molecular weight is 1000 to 10000.

Description

It occurs as a milky-white to light yellow, slightly viscous solid, and it is odorless or has a faint, characteristic odor.

It is practically insoluble in water, diethyl ether and ethanol, but slightly soluble in toluene and n-heptane.

Identification

Heat and compress this substance at 190°C to make a 50-100- μm film, and determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2850 cm^{-1} , 1460 cm^{-1} , 1380 cm^{-1} , 1156 cm^{-1} , 973 cm^{-1} and 730 cm^{-1} .

Purity

- (1) Clarity of solution

Dissolve 1 g of this substance in 100 mL of toluene at 80°C: the solution is clear.

- (2) Heavy metals: Not more than 10 ppm (Method 2)

Loss on drying: Not more than 1.0% (50 g, 160°C, 4 hours)

Residue on ignition: Not more than 0.1% (30 g, 900°C, 90 minutes)

Amorphous Propylene-Ethylene-Butene-1 Ternary Copolymer

Definition

Amorphous Propylene-Ethylene-Butene-1 Ternary Copolymer is a ternary copolymer of propylene, ethylene and butene-1. The mean molecular weight is 1000 to 10000.

Description

It occurs as a milky-white to light yellow, slightly viscous solid, and it is odorless or has a faint, characteristic odor.

It is practically insoluble in water, diethyl ether and ethanol, but slightly soluble in toluene and n-heptane.

Identification

Heat and compress this substance at 190°C to make a 50-100- μm film, and determine the infrared absorption

spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2850 cm^{-1} , 1460 cm^{-1} , 1380 cm^{-1} , 1156 cm^{-1} , 973 cm^{-1} , 760 cm^{-1} and 730 cm^{-1} .

Purity

(1) Clarity of solution

Dissolve 1 g of this substance in 100 mL of toluene at 80°C : the solution is clear.

(2) Heavy metals: Not more than 10 ppm (Method 2)

Loss on drying: Not more than 1.0% (50 g, 160°C , 4 hours)

Residue on ignition: Not more than 0.1% (30 g, 900°C , 90 minutes)

Amorphous Propylene-Butene-1 Copolymer

Definition

Amorphous Propylene-Butene-1 Copolymer is a copolymer of propylene and butene-1. The mean molecular weight is 1000 to 10000.

Description

It occurs as a milky-white to light yellow, slightly viscous solid, and it is odorless or has a faint, characteristic odor.

It is practically insoluble in water, diethyl ether and ethanol, but slightly soluble in toluene and n-heptane.

Identification

Heat and compress this substance at 190°C to make a 50-100- μm film, and determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2850 cm^{-1} , 1460 cm^{-1} , 1380 cm^{-1} , 1156 cm^{-1} , 973 cm^{-1} and 760 cm^{-1} .

Purity

(1) Clarity of solution

Dissolve 1 g of this substance in 100 mL of toluene at 80°C : the solution is clear.

(2) Heavy metals: Not more than 10 ppm (Method 2)

Loss on drying: Not more than 1.0% (50 g, 160°C , 4 hours)

Residue on ignition: Not more than 0.1% (30 g, 900°C , 90 minutes)

Amorphous Polypropylene Resin

Definition

Amorphous Polypropylene Resin is a polymer of propylene. The mean molecular weight is 1000 to 10000.

Description

It occurs as a milky-white to light yellow, slightly viscous solid, and it is odorless or has a faint, characteristic odor.

It is practically insoluble in water, diethyl ether and ethanol, but slightly soluble in toluene and n-heptane.

Identification

Heat and compress this substance at 190°C to make a 50-100- μm film, and determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2960 cm^{-1} , 2850 cm^{-1} , 1460 cm^{-1} , 1380 cm^{-1} , 1156 cm^{-1} and 973 cm^{-1} .

Purity

(1) Clarity of solution

Dissolve 1 g of this substance in 100 mL of toluene at 80°C: the solution is clear.

(2) Heavy metals: Not more than 10 ppm (Method 2)

Loss on drying: Not more than 1.0% (50 g, 160°C, 4 hours)

Residue on ignition: Not more than 0.1% (30 g, 900°C, 90 minutes)

Emulsion of Rosin Denatured with Fumaric Acid

Definition

Emulsion of Rosin Denatured with Fumaric Acid is an emulsion obtained by the emulsification of rosin denatured with fumaric acid with emulsifying agent.

Description

It is a white liquid and it is odorless or has a faint, characteristic odor.

Identification

Determine the infrared absorption spectrum of this substance, previously dried at 105 °C for about 2 hours, as directed in the film method under Infrared Spectrophotometry; it exhibits absorption at the wave number of about 1700 cm^{-1} .

pH: 4.0-6.5

Purity

(1) Heavy metals: Not more than 10 ppm (Method 2)

(2) Arsenic: Not more than 2 ppm (Method 2)

Aromatic Denatured Terpene Resin

Definition

Aromatic Denatured Terpene Resin is a synthetic resin obtained by hydrogenating a copolymer of terpene hydrocarbon compound and aromatic hydrocarbon compound having a substituent.

Description

It occurs as a light yellow, translucent, bead-like or flaky, easily-breakable solid, and it is practically odorless. It is freely soluble in chloroform and toluene, but practically insoluble in water and ethanol.

Identification

Dissolve about 1 g of this substance in 5 mL of chloroform, apply lightly this solution on the disk, evaporate the chloroform to make a film, and determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2900 cm^{-1} , 1600 cm^{-1} , 1450 cm^{-1} and 1375 cm^{-1} .

Acid value: Not more than 2.0 (Method 1)

Dissolve this substance in a mixture of toluene and ethanol (1:1). Use this solution for the test.

Heavy metals: Not more than 10 ppm (Method 2)

Loss on drying: Not more than 1% (1.0 g, 105°C, 4 hours)

Residue on ignition: Not more than 0.1% (10 g, 800°C)

Polyacrylamide Solution

Definition

Polyacrylamide Solution is a solution of copolymer of polyacrylamide.

Description

It is a light yellow, clear liquid, and it is odorless or has a faint, characteristic odor.

Identification

Determine the infrared absorption spectrum of this substance, previously dried at 105°C for about 2 hours, as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 3380 cm^{-1} , 1660 cm^{-1} , 1610 cm^{-1} (amide), 1460 cm^{-1} and 1130 cm^{-1} .

pH: 4.0-9.0

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Method 2)
- (3) Acrylic unreacted monomer: Not more than 1.5% (1.0 g)

Polyacrylamide-Polyvinyl Alcohol Copolymer Emulsion

Definition

Polyacrylamide-Polyvinyl Alcohol Copolymer Emulsion is a copolymer emulsion of polyvinyl alcohol and amide polyacrylate.

Description

It is a viscous, opaque liquid, and it is practically odorless.

Identification

- (1) Identification of polyvinyl alcohol

To 5 mL of this substance, add 1 drop of iodine TS: a dark blue or red color is produced. Separately, take 5 mL of this substance and add 10 mL of ethanol: a cotton-like precipitation is produced.

- (2) Identification of amide polyacrylate

Determine the infrared absorption spectrum of this substance, previously dried at 105°C for about 2 hours, as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 3380 cm^{-1} , 1660 cm^{-1} , 1610 cm^{-1} (amide), 1460 cm^{-1} and 1130 cm^{-1} .

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Method 2)
- (3) Acrylic unreacted monomer: Not more than 1.5% (1.0 g)

Polyester-Copolymer Polyester Bicomponent Fiber

Definition

Polyester-Copolymer Polyester Bicomponent Fiber is a polyester (polyethylene terephthalate)-core/copolymer polyester (polyethylene terephthalate/isophthalate copolymer polyester)-sheath bicomponent fiber.

Description

It occurs as colorless to white fibrous substances, and it is odorless.

Identification

- (1) Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 1720 cm^{-1} , 1580 cm^{-1} , 1500 cm^{-1} , 1410 cm^{-1} , 1260 cm^{-1} , 1100 cm^{-1} , 1015 cm^{-1} , 870 cm^{-1} and 725 cm^{-1} .
- (2) Place this substance near a flame. It melts and burns, and a black hard round ash remains.

Specific gravity: 1.37-1.38

Melting point: Polyester: 255°C - 260°C

Polyester copolymer: 110°C (softening point, observed with naked eye)

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Atomic Absorption Spectrophotometry)

Residue on ignition: Not more than 4% (Method 2)

Polyethylene Terephthalate Resin (PET)

Definition

Polyethylene Terephthalate Resin (PET) is a polyethylene terephthalate resin obtained by the esterification or transesterification of terephthalic acid or dimethyl terephthalate and ethylene glycol, followed by polycondensation.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 1720 cm^{-1} , 1580 cm^{-1} , 1250 cm^{-1} , 1100 cm^{-1} , 1015 cm^{-1} , 870 cm^{-1} and 725 cm^{-1} .

Specific gravity: 1.35-1.39

Melting point: 200 - 260°C

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Polyethylene Terephthalate Fiber

Definition

Polyethylene Terephthalate Fiber is a polyethylene terephthalate fiber obtained by the esterification or transesterification of terephthalic acid or dimethyl terephthalate and ethylene glycol, followed by polycondensation.

Description

It occurs as colorless to white fibrous substances, and it is odorless.

Identification

- (1) Determine the infrared absorption spectrum of this substance as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 1720 cm^{-1} , 1580 cm^{-1} , 1250 cm^{-1} , 1100 cm^{-1} , 1015 cm^{-1} , 870 cm^{-1} and 725 cm^{-1} .
- (2) Place this substance near a flame. It melts and burns, and a black hard round ash remains.

Specific gravity: 1.38-1.39

Melting point: 250-260°C

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Atomic Absorption Spectrophotometry)

Residue on ignition: Not more than 2.5% (Method 2)

Polyethylene Terephthalate/Polyethylene Bicomponent Fiber

Definition

Polyethylene Terephthalate/Polyethylene Bicomponent Fiber is a polyester (polyethylene terephthalate)-core/polyethylene-sheath bicomponent fiber.

Description

It occurs as colorless to white fibrous substances, and it is odorless.

Identification

Determine the infrared absorption spectrum as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2980 cm^{-1} , 2910 cm^{-1} , 1720 cm^{-1} , 1580 cm^{-1} , 1450 cm^{-1} , 1250 cm^{-1} , 1100 cm^{-1} , 1015 cm^{-1} , 870 cm^{-1} and 725 cm^{-1} .

Specific gravity: 1.07-1.37

Melting point: Polyester: 250-260°C

Polyethylene: 115-135°C

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Atomic Absorption Spectrophotometry)

Residue on ignition: Not more than 4% (Method 2)

Polyethylene Oxide

Definition

Polyethylene Oxide is a water-soluble polymer obtained by the ring-opening polymerization of ethylene oxide. The mean molecular weight is 2,000,000 to 10,000,000.

Description

It occurs as a white powder, and it is odorless or has a faint, characteristic odor.

Identification

Shake 0.2 g of this substance with 10 mL of water and 5 mL of ammonium thiocyanate-cobalt nitrate TS, and allow to stand: a blue color develops in the chloroform layer.

Viscosity

The viscosity of a solution (1 in 200) is 100-1000 mPa.s. (Brookfield type viscometer, No. 2, 12 rotations, 25°C, constant)

Purity

(1) Heavy metals: Not more than 20 ppm (Method 2)

(2) Arsenic: Not more than 2 ppm (Method 2)

Loss on drying: Not more than 4.0% (2.0 g, 105°C, 3 hours)

Residue on ignition: Not more than 5.0% (Method 1)

Polyethylene Resin

Definition

Polyethylene Resin is a polyethylene resin obtained by the polymerization of ethylene.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum as directed in the film method under Infrared Spectrophotometry: it exhibits absorption of polymer n-paraffin homologues.

Specific gravity: 0.85-1.00

Melting point: 90-140°C

Purity

(1) Clarity and color of solution

Dissolve 1.0 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.

(2) Heavy metals: Not more than 20 ppm (Method 2)

(3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0-g, Method 1)

Polyethylene Fiber

Definition

Polyethylene Fiber is a fiber of polyethylene obtained by the polymerization of ethylene.

Description

It occurs as colorless to white fibrous substances, and it is odorless.

Identification

- (1) Determine the infrared absorption spectrum as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2900 cm^{-1} , 1470 cm^{-1} , 1370 cm^{-1} , 740 cm^{-1} and 720 cm^{-1} .
- (2) Place this substance near a flame. It melts and burns with fumes emitting an odor of paraffin. A gray hard bead-like ash remains.

Specific gravity: 0.93-0.96

Melting point: 120-135°C

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 2.5% (Method 2)

Polyethylene/Polypropylene Bicomponent Fiber

Definition

Polyethylene/Polypropylene Bicomponent Fiber is a polypropylene-core/polyethylene-sheath or side-by-side bicomponent fiber.

Description

It occurs as colorless to white fibrous substances, and it is odorless.

Identification

- (1) Determine the infrared absorption spectrum as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2980 cm^{-1} , 2930 cm^{-1} , 2830 cm^{-1} , 1465 cm^{-1} , 1455 cm^{-1} , 1375 cm^{-1} , 1255 cm^{-1} , 1165 cm^{-1} , 995 cm^{-1} , 970 cm^{-1} , 840 cm^{-1} , 810 cm^{-1} , 740 cm^{-1} and 725 cm^{-1} .
- (2) Place this substance near a flame. It melts and burns with fumes emitting an odor of paraffin. A gray hard bead-like ash remains.

Specific gravity: 0.91-1.01

Melting point: Polypropylene: 160-170°C

Polyethylene: 115-135°C

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 4% (Method 2)

Polyvinyl Chloride Fiber (PVC Fiber)

Definition

Polyvinyl Chloride Fiber (PVC Fiber) is a fiber of polyvinyl chloride obtained by the suspension

polymerization of vinyl chloride.

Description

It occurs as colorless to white fibrous substances, and it is odorless.

Identification

- (1) Determine the infrared absorption spectrum of this substance as directed in the potassium bromide disk method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2950 cm^{-1} , 1420 cm^{-1} , 1240 cm^{-1} , 1070 cm^{-1} , 960 cm^{-1} and 700 cm^{-1} .
- (2) When burned, it softens and shrinks with fume and becomes a black block coal.

Specific gravity: 1.39

Melting point: 200-210°C

Purity

Vinyl chloride

Take 1.0 g of this substance in a 20-mL volumetric flask. Add about 10 mL of tetrahydrofuran for gas chromatography, dissolve by occasional shaking in a cold place, add tetrahydrofuran for gas chromatography, previously cooled, to make 20 mL while cooling, and use this solution as the sample solution. Perform the test with 2 μL each of the sample solution and Standard Vinyl Chloride Solution as directed under Gas Chromatography according to the following conditions. Determine the peak heights, H_t and H_s , of vinyl chloride of respective solutions: H_t is not higher than H_s .

Operating conditions

Detector: Hydrogen flame-ionization detector

Column: A column about 3 mm in inside diameter and 2 or 3 m in length, packed with siliceous earth for gas chromatography (150 to 180 μm) coated with polypropylene glycol for gas chromatography in 10-15%

Column temperature: A constant temperature of about 60-70°C

Carrier gas: Nitrogen

Flow rate: Adjust the flow rate so that the retention time of vinyl chloride is about 1.5 minutes.

Selection of column: Proceed with 2 μL of Standard Vinyl Chloride Solution under the above operating conditions. Use a column from which vinyl chloride and ethanol are eluted in that order, with a good resolution between their peaks.

Detection sensitivity: Adjust it so that the peak height of vinyl chloride obtained from 2 μL of the Standard Vinyl Chloride Solution is 50 to 70 mm.

Loss on drying: Not more than 1.0% (1.0 g, 105°C, 2 hours)

Residue on ignition: Not more than 2.5% (Method 2)

Polyvinyl Alcohol

Definition

Polyvinyl Alcohol is a polymer obtained by saponifying polyvinyl acetate and is expressed as $-\text{[CH}_2\text{-CHOH]}_n\text{-[CH}_2\text{-CHOCOCH}_3\text{]}_m\text{-}$. The viscosity of this substance is expressed as mPa·s. Usually, it is

between 2 mPa·s and 100 mPa·s.

Description

It occurs as colorless to pale yellowish white granules, powder or fibrous substances, and it is odorless or has a faint odor of acetic acid.

It is practically insoluble in ethanol, diethyl ether, and chloroform.

To this substance add water, and heat: A clear, viscous solution is obtained.

It is hygroscopic.

Identification

- (1) Dissolve 0.5 g of this substance in 10 mL of water by heating, cool, add 1 drop of iodine TS to 5 mL of this solution and allow to stand: a dark red to blue color develops.
- (2) Dissolve 0.01 g of this substance in 100 mL of water by heating, cool, add 1 drop of iodine TS to 5 mL of this solution, mix, and add 5 mL of a solution of boric acid (1 in 25): a blue color develops.
- (3) To 2 mL of the solution obtained in (1), add 5 mL of ethanol: a white cotton-like precipitate is produced.

Viscosity: 85-115% of the labeled value (mPa·s)

Take 4.000 g of this substance, previously dried, add 95 mL of water, allow to stand for 30 minutes and dissolve by heating under a reflux condenser for 2 hours while stirring. After cooling, add water to make 100.0 g, and mix. Allow to stand still to remove bubbles and perform the test at $20 \pm 0.1^\circ\text{C}$ as directed in Method 1.

pH: 5.0 - 8.0 (1 in 25)

Saponification value: Not less than 70 mol%.

Weigh accurately the amount as directed in Table 1 according to the estimated saponification value, previously dried, transfer into a glass-stoppered conical flask, add 100 mL of water, and dissolve by heating while stirring for 2 hours. After cooling, add exactly 25 mL of 0.1 mol/L or 0.5 mol/L sodium hydroxide VS according to Table 1, stopper tightly, and allow to stand for 2 hours. Then add exactly 25 mL of sulfuric acid at the same concentration as that of sodium hydroxide VS, shake well, and titrate with 0.1 mol/L or 0.5 mol/L sodium hydroxide VS according to Table 1 (indicator: 3 drops of phenolphthalein TS). Perform a blank determination in the same manner.

$$\text{Saponification value (mol\%): } 100 - \frac{44.05A}{60.05-0.42A}$$

$$A = \frac{0.6005 \times (a-b)FD}{\text{Amount (g) of sample}}$$

a: Volume (mL) of 0.1 mol/L or 0.5 mol/L sodium hydroxide VS consumed

b: Volume (mL) of 0.1 mol/L or 1.0 mol/L sodium hydroxide VS consumed in the blank determination

F: Molarity factor of 0.1 mol/L or 0.5 mol/L sodium hydroxide VS

D: Concentration of sodium hydroxide VS (0.1 mol/L or 0.5 mol/L)

Table 1 Estimated saponification value, and amount of the sample and specified solution for use

Estimated saponification value mol%	Amount of sample G	Solution specified for use	
		Concentration mol/L	Used amount mL
Not less than 97	3	0.1	25.00
Not less than 90, less than 97	3	0.5	25.00
Not less than 80, less than 90	2	0.5	25.00
Not less than 70, less than 80	1	0.5	25.00

Purity

- (1) Clarity and color of solution

To 1.0 g of this substance, add 20 mL of water, disperse by stirring well, heat for not less than 2 hours while stirring and cool: the solution is colorless and clear.

- (2) Heavy metals: Not more than 10 ppm (2.0 g, Method 2, Standard Lead Solution 2.0 mL)
(3) Arsenic: Not more than 2 ppm (Method 2)

Loss on drying: Not more than 6.0% (1.0 g, 105°C, 3 hours)

Residue on ignition: Not more than 2% (Method 1)

Polypropylene Copolymer Fiber

Definition

Polypropylene Copolymer Fiber is a fiber of propylene-ethylene copolymer obtained by copolymerizing propylene and ethylene.

Description

It occurs as colorless to white fibrous substances, and it is odorless.

Identification

Determine the infrared absorption spectrum as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2950 cm⁻¹, 2920 cm⁻¹, 2830 cm⁻¹, 1455 cm⁻¹, 1375 cm⁻¹, 1255 cm⁻¹, 1165 cm⁻¹, 970 cm⁻¹, 840 cm⁻¹ and 720 cm⁻¹.

Specific gravity: 0.89 - 0.90

Melting point: 148°C

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
(2) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 4% (Method 2)

Polypropylene/Copolymer Polypropylene Bicomponent Fiber

Definition

Polypropylene/Copolymer Polypropylene Bicomponent Fiber is a polypropylene-core/polypropylene copolymer (propylene-ethylene copolymer)-sheath or side-by-side bicomponent fiber.

Description

It occurs as colorless to white fibrous substances, and it is odorless.

Identification

- (1) Determine the infrared absorption spectrum as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2980 cm^{-1} , 2940 cm^{-1} , 2830 cm^{-1} , 1460 cm^{-1} , 1380 cm^{-1} , 1255 cm^{-1} , 1165 cm^{-1} , and 710 cm^{-1} .
- (2) Place this substance near a flame. It melts and burns with fumes emitting an odor of paraffin. A gray hard bead-like ash remains.

Specific gravity: 0.91-0.94

Melting point: Polypropylene: 160-170°C

Copolymer polypropylene: 115-148°C

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 4% (Method 2)

Polypropylene Resin (PP)

Definition

Polypropylene Resin (PP) is a polypropylene resin obtained by polymerizing propylene.

Description

It occurs as translucent powder or granules, and it is practically odorless.

Identification

Determine the infrared absorption spectrum as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2930 cm^{-1} , 2830 cm^{-1} , 1455 cm^{-1} , 1375 cm^{-1} , 1255 cm^{-1} , 1165 cm^{-1} , 995 cm^{-1} , 970 cm^{-1} , 840 cm^{-1} and 810 cm^{-1} .

Specific gravity: 0.89-0.94

Melting point: 150-170°C

Purity

- (1) Clarity and color of solution
Dissolve 1 g of this substance in 50 mL of xylene by heating: the solution is colorless and clear.
- (2) Heavy metals: Not more than 20 ppm (Method 2)
- (3) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 0.1% (5.0 g, Method 1)

Polypropylene Fiber

Definition

Polypropylene Fiber is a fiber obtained by the polymerization of propylene.

Description

It occurs as colorless to white fibrous substances, and it is odorless.

Identification

- (1) Determine the infrared absorption spectrum as directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2930 cm^{-1} , 2830 cm^{-1} , 1455 cm^{-1} , 1375 cm^{-1} , 1255 cm^{-1} , 1165 cm^{-1} , 995 cm^{-1} , 970 cm^{-1} , 840 cm^{-1} and 810 cm^{-1} .
- (2) Place this substance near a flame. It melts and burns with fumes emitting an odor of paraffin. A gray hard bead-like ash remains.

Specific gravity: 0.89-0.94

Melting point: 160-170°C

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Method 2)

Residue on ignition: Not more than 2.5% (Method 2)

Solution of Petroleum Resin Denatured with Maleic Acid**Definition**

Solution of Petroleum Resin Denatured with Maleic Acid is obtained by adding an aqueous solution of potassium hydroxide to warmed maleinized petroleum resin and maleinized rosin, followed by neutralization while well stirring, then emulsification and dispersion by adding water, cooling and filtration.

Description

It is a pale yellow-brown, translucent liquid, and it has a characteristic odor.

Identification

Dissolve 1.0 g of this substance in 5 mL of water, neutralize with 0.2 mL of hydrochloric acid, add 10 mL of diethyl ether and stir. Take the ether layer, evaporate the solvent and determine the infrared absorption spectrum as directed in the potassium bromide disk method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 1860 cm^{-1} , 1780 cm^{-1} , 1700 cm^{-1} , 720 cm^{-1} and 700 cm^{-1} .

pH: 9.5-10.5 (1 in 6)

Purity

- (1) Heavy metals: Not more than 10 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Method 2)

Solution of Rosin Denatured with Maleic Acid**Definition**

Solution of Rosin Denatured with Maleic Acid is an aqueous solution of the alkali metal salt of rosin denatured with maleic acid.

Description

It is a brown, clear liquid, and it has a characteristic odor of rosin.

Identification

Determine the infrared absorption spectrum of this substance, previously dried at 105 °C for about 2 hours, as

directed in the film method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about between 3000 cm^{-1} and 2800 cm^{-1} , 1570 cm^{-1} , and 700 cm^{-1} .

pH: 9.0-11.0

Purity

- (1) Heavy metals: Not more than 10 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Method 2)

α -Methylstyrene-group Resin

Definition

α -Methylstyrene-group Resin is an oligomer obtained by the polymerization of α -methylstyrene monomer (50-90%) and styrene monomer (10-50%) using boron trifluoride as a catalyst. The mean molecular weight is 600 to 5000.

Description

It occurs as a white, slightly viscous solid, and it is freely soluble in acetone and toluene, but insoluble in water and methanol.

Identification

Dissolve about 4.0 g of this substance in 100 mL of carbon tetrachloride and inject the solution into a fixed cell of sodium chloride. Perform the test as directed in the Solution method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2970 cm^{-1} and 2930 cm^{-1} .

Purity

- (1) Clarity of solution

To 1 g of this substance, add 100 mL of toluene and heat on a water bath: the solution is clear.

- (2) Heavy metals: Not more than 50 ppm (0.5 g, Method 2, Standard Lead Solution 2.5 mL)

Loss on drying: Not more than 1.0% (1.0 g, 105°C , 4 hours)

Residue on ignition: Not more than 0.1% (1.0 g, $450\text{-}550^{\circ}\text{C}$)

Flocculent Pulp

Definition

Flocculent Pulp is a flocculent chemical pulp.

Description

- (1) It is white in color, odorless, and contains no foreign matter.
- (2) It does not remarkably contain fiber mass.

Purity

- (1) Lignin

Dissolve 0.1 g of phloroglucin in 15 mL of hydrochloric acid, add water to make 20 mL and drop onto this substance: no marked pink or red color develops.

- (2) Coloring matter

Immerse 10 g of this substance in 100 mL of ethanol, press out, transfer 50 mL of the extract into a

Nessler tube and observe downward: a yellow color may develop but neither blue nor green color develops.

(3) Acidity or alkalinity

To 10 g of this substance, add 100 mL of freshly boiled and cooled water and allow to cool. To 25 mL of the solution, add 3 drops of phenolphthalein TS: no red color develops. Separately, to 25 mL of the same solution, add 1 drop of methyl orange TS: no red color develops.

(4) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows neither marked fluorescence nor fluorescence by contamination.

(5) Sedimentation velocity

Prepare a test basket, weighing about 3 g from copper wire 0.4 mm in diameter (No. 26) in the form of a cylinder 50 mm in diameter and 80 mm in depth, with 20-mm intervals between the wires. Place 5.0 g of this substance in the test basket, drop the basket on its side gently into the water about 200 mm in depth at ordinary temperature from the height of about 10 mm above the water surface: the basket sinks in water within 8 seconds.

Total ash: Not more than 0.65% (5.0 g)

Sorbitan Monolaurate

Definition

Sorbitan Monolaurate consists mainly of laurate monoester of sorbitan.

Description

It is a pale yellow to yellow-brown liquid, and it has a faint, characteristic odor.

Identification

- (1) To 0.5 g of this substance, add 5 mL of ethanol, dissolve by heating on a water bath, add 5 mL of dilute sulfuric acid, and heat further for 30 minutes and cool: oily drops or a white to yellowish white solid is precipitated. This separated oily drops or solid dissolves when shaken with 5 mL of diethyl ether.
- (2) Shake 2 mL of the separately oily drops or solid in (1) with 2 mL of freshly prepared catechol solution (1 in 10), then with 5 mL of sulfuric acid: a red to red-brown color develops.
- (3) Saponify 5 g of this substance using the saponification method and completely evaporate ethanol. Dissolve the residue in 50 mL of water, acidify with hydrochloric acid (indicator: methyl orange TS), and extract the residue with 30 mL of diethyl ether twice. Combine the ether layers, wash with 20 mL portions of water until the washings become neutral, and evaporate the ether on a water bath: the acid value of the residue is between 260 to 280 (0.5 g, Method 1). Use 50 mL of the 0.5 mol/L ethanol solution of potassium hydroxide for saponification.

Acid value: Not more than 13 (2.0 g, Method 2)

Saponification value: 155-174

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)

(2) Arsenic: Not more than 2 ppm (Method 2)

Loss on drying: Not more than 3.0% (5.0 g, 105°C, 1 hour)

Residue on ignition: Not more than 1.0% (3.0 g, Method 3)

Cotton

Definition

Cotton is cotton wool adherent to seeds of raw cotton.

Description

- (1) It is white in color, odorless, and contains no foreign matter.
- (2) It does not remarkably contain broken pieces of pericarp and seed, or nep.

Identification

It is soluble in ammonium copper TS and insoluble in ethanol.

Purity

- (1) Coloring matter

Immerse 10 g of this substance in 100 mL of ethanol, press out, transfer 50 mL of the extract into a Nessler tube and observe downward: a yellow color may develop but neither blue nor green color develops.

- (2) Acidity or alkalinity

To 10 g of this substance, add 100 mL of freshly boiled and cooled water and allow to cool. To 25 mL of the solution, add 3 drops of phenolphthalein TS: no red color develops. Separately, to 25 mL of the same solution, add 1 drop of methyl orange TS: no red color develops.

- (3) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows neither marked fluorescence nor fluorescence by contamination.

Total ash: Not more than 0.25% (5.0 g)

Aluminum Sulfate (Solution)

Definition

Aluminum Sulfate (Solution) is an aqueous solution containing not less than 8.0% and not more than 8.2% of aluminum sulfate $[\text{Al}_2(\text{SO}_4)\cdot 18\text{H}_2\text{O}]$.

Description

It is a colorless to light yellow-brown, clear liquid, and it is odorless.

Identification

- (1) It responds to the Qualitative Tests for aluminum salt.
- (2) It responds to the Qualitative Tests (1) and (2) for sulfate.

pH: 3.0-4.0 (2 w/v% solution of this substance)

Purity

- (1) Iron

Transfer 1.0 g of this substance into a Nessler tube, add 6 mL of dilute nitric acid and water to make 20 mL. Add 0.05 g of ammonium persulfate and 5 mL of ammonium thiocyanate TS, shake, add 15 mL of n-butanol and shake vigorously for 30 seconds: the n-butanol layer has no more color than the following control solution.

Control solution: Using 2.0 mL of Standard Iron Solution instead of this substance, perform the test in the same manner.

- (2) Heavy metals: Not more than 10 ppm (Method 1)
- (3) Arsenic: Not more than 2 ppm (0.40 g, Method 1)

Flocculent Rayon

Definition

Flocculent Rayon is flocculent regenerated fiber made from plant-based fibers.

Description

It is white in color, odorless, and contains no foreign matter.

Identification

It is soluble in sulfuric acid. It swells with ammonium copper TS and then dissolves.

Purity

- (1) Coloring matter

Immerse 10 g of this substance in 100 mL of ethanol, press out, transfer 50 mL of the extract into a Nessler tube and observe downward: a yellow color may develop but neither blue nor green color develops.

- (2) Acidity or alkalinity

To 10 g of this substance, add 100 mL of freshly boiled and cooled water and allow to cool. To 25 mL of the solution, add 3 drops of phenolphthalein TS: no red color develops. Separately, to 25 mL of the same solution, add 1 drop of methyl orange TS: no red color develops.

- (3) Fluorescence

Irradiate this substance with ultraviolet rays (main wavelength: 365 nm) in a dark place: it shows neither marked fluorescence nor fluorescence by contamination.

- (4) Sedimentation velocity

Prepare a test basket, weighing about 3 g from copper wire 0.4 mm in diameter (No. 26) in the form of a cylinder 50 mm in diameter and 80 mm in depth, with 20-mm intervals between the wires. Place 5.0 g of this substance in the test basket, drop the basket on its side gently into the water about 200 mm in depth at ordinary temperature from the height of about 10 mm above the water surface: the basket sinks in water within 8 seconds..

Total ash: Not more than 0.25% (5.0 g)

Not more than 1.2% (5.0 g) (delustered)

Rayon Fiber

Definition

Rayon Fiber is cellulose fiber obtained by regenerating cellulose using the viscose method.

Description

It occurs as colorless to light yellow fibrous substances, and it is practically odorless.

Identification

- (1) Determine the infrared absorption spectrum as directed in the potassium bromide disk method under Infrared Spectrophotometry: it exhibits absorption at the wave numbers of about 2900 cm^{-1} , 1650 cm^{-1} and 890 cm^{-1} .
- (2) Burn this substance: it emits an odor of burning paper, and the residual ash is thin and has a black or gray color.
- (3) It is soluble in ammonium copper TS.

Specific gravity: 1.50-1.52

Melting point: 260-300°C (with decomposition)

Purity

- (1) Heavy metals: Not more than 20 ppm (Method 2)
- (2) Arsenic: Not more than 2 ppm (Method 2)

Loss on drying: Not more than 11.0% (2.0 g, 105°C, 3 hours)

Residue on ignition: Not more than 2.5% (Method 2)

Part 2 - Colorants

Silicon Dioxide

C.I. Acid Blue 9 (Blue No. 205)

C.I. Acid Blue 74 (Blue No. 2)

C.I. Acid Red 51 (Red No. 3)

C.I. Direct Yellow 12

C.I. Direct Orange 26

C.I. Direct Violet 51

C.I. Direct Blue 1

C.I. Direct Blue 86

C.I. Direct Blue 106

C.I. Direct Blue 203

C.I. Direct Red 23

C.I. Direct Red 31

C.I. Direct Red 80

C.I. Direct Red 81

C.I. Direct Red 227

C.I. Vat Blue 1 (Blue No. 201)

C.I. Pigment Yellow 1 (Yellow No. 401)

C.I. Pigment Yellow 12 (Yellow No. 205)

C.I. Pigment Yellow 14

C.I. Pigment Yellow 83

C.I. Pigment Orange 13 (Orange No. 204)

C.I. Pigment Green 7

C.I. Pigment Violet 19

C.I. Pigment Violet 23

C.I. Pigment Blue 15 (Blue No. 404)

C.I. Pigment Blue 27 (Iron Blue)

C.I. Pigment Brown 6 (Iron Oxide Brown)

C.I. Pigment Brown 24 (Chrome Titan Yellow)

C.I. Pigment Black 7 (Carbon Black)

C.I. Pigment White 4 (Zinc Oxide)

C.I. Pigment White 6 (Titanium Dioxide)

C.I. Pigment White 18 (Calcium Carbonate)

C.I. Pigment White 19 (Kaolin)

C.I. Pigment White 21 (Barium Sulfate)

C.I. Pigment Red 22 (Red No. 404)

C.I. Pigment Red 48 (Red No. 405)

C.I. Pigment Red 57 (Red No. 201)

C.I. Pigment Red 57-1 (Red No. 202)

C.I. Pigment Red 166

C.I. Food Blue 2 (Blue No.1)

C.I. Basic Violet 3

C.I. Reactive Orange 16

C.I. Reactive Black 5

C.I. Reactive Blue 21

C.I. Reactive Blue 27

C.I. Reactive Blue 28

C.I. Reactive Blue 38

C.I. Reactive Red 21