

a previously dried and tared evaporation dish. The difference between the mass of the residue and the mass obtained from a blank determination does not exceed 15.0 mg.

(3) Heavy metals—Proceed with 2.0 g of Powdered Cellulose according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard lead Solution (not more than 10 ppm).

Loss on drying Not more than 6.0% (1 g, 105°C, 2 hours).

Residue on ignition Not more than 0.3% (1 g calculated on the dried basis, the addition of sulfuric acid being omitted from the procedure).

Microbial limits The total aerobic microbial count does not exceed 1000 per g, the total combined fungus and yeast count does not exceed 100 per g, and *Escherichia coli*, *Salmonella* species, *Staphylococcus aureus* and *Pseudomonas aeruginosa* are not observed.

Containers and storage Containers—Tight containers.

Cellulose Acetate Phthalate

酢酸フタル酸セルロース

Cellulose acetate benzene-1,2-dicarboxylate
[9004-38-0]

Cellulose Acetate Phthalate is a reaction product of phthalic anhydride and partially acetylated cellulose.

Cellulose Acetate Phthalate, calculated on the anhydrous and free acid-free basis, contains not less than 21.5% and not more than 26.0% of acetyl group ($-\text{COCH}_3$: 43.05), and not less than 30.0% and not more than 40.0% of carboxybenzoyl group ($-\text{COC}_6\text{H}_4\text{COOH}$: 149.13).

Description Cellulose Acetate Phthalate occurs as a white powder or grain. It is odorless or has a faint, acetous odor.

It is freely soluble in acetone, and practically insoluble in water, in methanol, in ethanol (95) and in dichloromethane.

Identification (1) Determine the infrared absorption spectrum of Cellulose Acetate Phthalate as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum or spectrum of Cellulose Acetate Phthalate Reference Standard: both spectra exhibit similar intensities of absorption at the same wave numbers.

(2) Dissolve 150 mg of Cellulose Acetate Phthalate in 1 mL of acetone, and pour on a surface of a transparent glass plate in a well-ventilated place: a lustrous transparent film is formed after evaporating of the acetone.

Viscosity Weigh accurately a quantity of Cellulose Acetate Phthalate, equivalent to 15 g calculated on the anhydrous basis, dissolve in 85 g of a mixture of acetone and water (249: 1 in mass), and perform the test with this solution at $25 \pm 0.2^\circ\text{C}$ as directed in Method 1 under the Viscosity Determination to obtain the kinematic viscosity ν . Separately, determine the density, ρ , of Cellulose Acetate Phthalate as directed under the Determination of Specific Gravity and Density,

and calculate the viscosity, η , as $\eta = \rho\nu$: not less than 45 mPa·s and not more than 90 mPa·s.

Purity (1) Heavy metals—Proceed with 2.0 g of Cellulose Acetate Phthalate according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

(2) Free acids—Weigh accurately about 3.0 g of Cellulose Acetate Phthalate, put in a glass-stoppered conical flask, add 100 mL of diluted methanol (1 in 2), stopper tightly, and filter after shaking for 2 hours. Wash both the flask and residue with two 10-mL portions each of diluted methanol (1 in 2), combine the washes to the filtrate, and titrate with 0.1 mol/L sodium hydroxide VS (indicator: 3 drops of phenolphthalein TS). Perform the blank determination with 120 mL of diluted methanol (1 in 2), and make any necessary correction.

$$\text{Amount (\%)} \text{ of free acids} = \frac{0.8306 \times A}{W}$$

A: amount (mL) of 0.1 mol/L sodium hydroxide consumed

W: amount (g) of the test sample, calculated on the anhydrous basis

The amount of free acids is not more than 3.0%, calculated as phthalic acid ($\text{C}_8\text{H}_6\text{O}_4$: 166.13).

Water Not more than 5.0% (1 g, direct titration, using a mixture of dehydrated methanol and dichloromethane (3:2) instead of methanol for Karl Fischer method).

Residue on ignition Not more than 0.1% (1 g).

Assay (1) Carboxybenzoyl group—Weigh accurately about 1 g of Cellulose Acetate Phthalate, dissolve in 50 mL of a mixture of ethanol (95) and acetone (3: 2), and titrate with 0.1 mol/L sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS). Perform a blank determination, and make any necessary correction.

Content (%) of carboxybenzoyl group ($\text{C}_8\text{H}_5\text{O}_3$)

$$= \frac{\frac{1.491 \times A}{W} - 1.795 \times B}{100 - B} \times 100$$

A: amount (mL) of 0.1 mol/L sodium hydroxide consumed

B: amount (%) of free acids obtained in Purity (2) Free acids

W: amount (g) of the test sample, calculated on the anhydrous basis

(2) Acetyl group—Weigh accurately about 500 mg of Cellulose Acetate Phthalate, put in a glass-stoppered conical flask, add 50 mL of water and exactly 50 mL of 0.5 mol/L sodium hydroxide VS, and boil for 60 minutes under a reflux condenser. After cooling, add 5 drops of phenolphthalein TS, and titrate with 0.5 mol/L hydrochloric acid VS. Perform a blank determination, and make any necessary correction.

Content (%) of free acids and bound acetyl group ($\text{C}_2\text{H}_3\text{O}$)

$$= \frac{2.152 \times A}{W}$$

A: amount (mL) of 0.5 mol/L sodium hydroxide consumed

W: amount (g) of the test sample, calculated on the anhydrous basis

$$\begin{aligned} &\text{Content (\% of acetyl group (C}_2\text{H}_3\text{O)} \\ &= \frac{100 \times (P - 0.5182 \times B)}{100 - B} \times -0.5772 \times C \end{aligned}$$

B: amount (%) of free acids obtained in Purity (2) Free acids

C: content (%) of carboxybenzoyl group

P: content (%) of free acids and bound acetyl group (C₂H₃O)

Containers and storage Containers—Tight containers.

Cetanol

セタノール

Cetanol is a mixture of solid alcohols, and consists chiefly of C₁₆H₃₄O.

Description Cetanol occurs as unctuous, white flakes, granules, or masses. It has a faint, characteristic odor. It is tasteless.

It is very soluble in pyridine, freely soluble in ethanol (95), in ethanol (99.5) and in diethyl ether, very slightly soluble in acetic anhydride, and practically insoluble in water.

Melting point 47 – 53°C Prepare the sample according to Method 2, then attach tightly a capillary tube to the bottom of the thermometer by means of a rubber band or by any suitable means, and make the bottom of the capillary tube equal in position to the lower end of the thermometer. Insert this thermometer into a test tube 17 mm in inside diameter and about 170 mm in height, fasten the thermometer with cork stopper so that the lower end of the thermometer is about 25 mm distant from the bottom of the test tube. Suspend the test tube in a beaker containing water, and heat the beaker with constant stirring until the temperature rises to 5°C below the expected melting point. Then regulate the rate of increase to 1°C per minute. The temperature at which the sample is transparent and no turbidity is produced is taken as the melting point.

Acid value Not more than 1.0.

Ester value Not more than 2.0.

Hydroxyl value 210 – 232

Iodine value Not more than 2.0.

Purity (1) Clarity of solution—Dissolve 3.0 g of Cetanol in 25 mL of ethanol (99.5) by warming; the solution is clear.

(2) Alkali—To the solution obtained in (1) add 2 drops of phenolphthalein TS: no red color develops.

Residue on ignition Not more than 0.05% (2 g).

Containers and storage Containers—Well-closed containers.

Chlorinated Lime

サラン粉

Chlorinated Lime contains not less than 30.0% of available chlorine (Cl: 35.45).

Description Chlorinated Lime occurs as a white powder. It has a chlorine-like odor.

It dissolves partially in water. The solution changes red litmus paper to blue, then gradually decolorizes.

Identification (1) To Chlorinated Lime add dilute hydrochloric acid: a gas, which has the odor of chlorine, evolves, and the gas changes moistened starch-potassium iodide paper to blue.

(2) Shake 1 g of Chlorinated Lime with 10 mL of water, and filter: the filtrate responds to the Qualitative Tests (2) and (3) for calcium salt.

Assay Weigh accurately about 5 g of Chlorinated Lime, transfer to a mortar, and triturate thoroughly with 50 mL of water. Transfer to a 500-mL volumetric flask with the aid of water, and add water to make 500 mL. Mix well, immediately take exactly 50 mL of the mixture in an iodine flask, add 10 mL of potassium iodide TS and 10 mL of dilute hydrochloric acid, and titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 3 mL of starch TS). Perform a blank determination, and make any necessary correction.

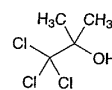
$$\begin{aligned} &\text{Each mL of 0.1 mol/L sodium thiosulfate VS} \\ &= 3.5453 \text{ mg of Cl} \end{aligned}$$

Containers and storage Containers—Tight containers.

Storage—Light-resistant, and in a cold place.

Chlorobutanol

クロロブタノール



C₄H₇Cl₃O: 177.46

1,1,1-Trichloro-2-methylpropan-2-ol [57-15-8]

Chlorobutanol contains not less than 98.0% of C₄H₇Cl₃O, calculated on the anhydrous basis.

Description Chlorobutanol occurs as colorless or white crystals. It has a camphoraceous odor.

It is very soluble in methanol, in ethanol (95) and in diethyl ether, and slightly soluble in water.

It slowly volatilizes in air.

Melting point: not lower than about 76°C.

Identification (1) To 5 mL of a solution of Chlorobutanol (1 in 200) add 1 mL of sodium hydroxide TS, then slowly add 3 mL of iodine TS: a yellow precipitate is produced and the odor of iodoform is perceptible.