dard, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry: both spectra exhibit similar intensities of absorption at the same wave numbers. If any difference appears, dissolve the sample and the Reference Standard in ethyl acetate, evaporate to dryness, and repeat the test on the residues.

Content uniformity Transfer 1 tablet of Prednisolone Tablets to a volumetric flask, and shake with 10 mL of water until the tablet is disintegrated. Add 50 mL of methanol, shake for 30 minutes, and add methanol to make exactly 100 mL. Centrifuge this solution, pipet x mL of the supernatant liquid, and add methanol to make exactly V mL to provide a solution that contains about 10 μg of prednisolone ($C_{21}H_{28}O_5$) per ml, and use this solution as the sample solution. Separately, weigh accurately about 0.010 g of Prednisolone Reference Standard, previously dried at 105°C for 3 hours, dissolve in 10 mL of water and 50 mL of methanol, and add methanol to make exactly 100 mL. Pipet 5 mL of this solution, add methanol to make exactly 50 mL, and use this solution as the standard solution. Determine the absorbances, A_T and A_S, of the sample solution and the standard solution at 242 nm as directed under the Ultraviolet-visible Spectrophotometry.

Amount (mg) of prednisolone ($C_{21}H_{18}O_5$)
= amount (mg) of Prednisolone Reference Standard
$\times \frac{A_T}{A_S} \times \frac{V}{10} \times \frac{1}{x}$

Dissolution test Take 1 tablet of Prednisolone Tablets, perform the test as directed in Method 2 under the Dissolution Test at 100 revolutions per minute using 900 mL of water as the test solution. Twenty minutes after the start of the test, take 20 mL or more of the dissolved solution, and filter through a membrane filter with pore size of 0.8 μm or less. Discard the first 10 mL of the filtrate, and use the subsequent filtrate as the sample solution. Weigh accurately about 0.010 g of Prednisolone Reference Standard, previously dried at 105°C for 3 hours, and dissolve in ethanol (95) to make exactly 100 mL. Pipet 5 mL of this solution, add water to make exactly 100 mL, and use this solution as the standard solution. Determine the absorbances, A_T and A_S, of the sample solution and the standard solution at the maximum wavelength at about 242 nm as directed under the Ultraviolet-visible Spectrophotometry.

The dissolution rate of Prednisolone Tablets after 20 minutes should be not less than 70%.

Dissolution rate (%) with respect to
the labeled amount of prednisolone ($C_{21}H_{28}O_5$)
$= W_S \times \frac{A_T}{A_S} \times \frac{45}{C}$

W_S: Amount (mg) of Prednisolone Reference Standard.
C: Labeled amount (mg) of prednisolone ($C_{21}H_{28}O_5$) in 1 tablet.

Assay Weigh accurately and powder not less than 20 Prednisolone Tablets using an agate mortar. Weigh accurately a portion of the powder, equivalent to about 5 mg of prednisolone ($C_{21}H_{28}O_5$), add 1 mL of water, and shake gently. Add exactly 5 mL of the internal standard solution and 15 mL of methanol, and shake vigorously for 20 minutes. To 1 mL of this solution add the mobile phase to make 10 mL, and filter through a membrane filter with pore size of 0.45 μm. Discard the first 3 mL of the filtrate, and use the subsequent filtrate as the sample solution. Separately, weigh accurately about 0.025 g of Prednisolone Reference Standard, previously dried at 105°C for 3 hours, dissolve in 50 mL of methanol, add exactly 25 mL of the internal standard solution, and add methanol to make 100 mL. To 1 mL of this solution add the mobile phase to make 10 mL, and use this solution as the standard solution. Proceed as directed in the Assay under Prednisolone with these solutions.

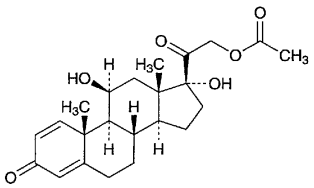
Amount (mg) of prednisolone ($C_{21}H_{28}O_5$)
= amount (mg) of Prednisolone Reference Standard
$\times \frac{Q_T}{Q_S} \times \frac{1}{5}$

Internal standard solution—A solution of methyl parahydroxybenzoate in methanol (1 in 2000).

Containers and storage Containers—Tight containers.

Prednisolone Acetate

酢酸プレドニゾロン



$C_{23}H_{30}O_6$: 402.48
11β,17,21-Trihydroxypregna-1,4-diene-3,20-dione 21-acetate [*52-21-1*]

Prednisolone Acetate, when dried, contains not less than 96.0% and not more than 102.0% of $C_{23}H_{30}O_6$.

Description Prednisolone Acetate occurs as a white, crystalline powder.

It is slightly soluble in methanol, in ethanol (95), in ethanol (99.5), and in chloroform, and practically insoluble in water.

Melting point: about 235°C (with decomposition).

Identification (1) To 2 mg of Prednisolone Acetate add 2 mL of sulfuric acid, and allow to stand for 2 to 3 minutes: a deep red color, without fluorescence, develops. To this solution add 10 mL of water cautiously: the color disappears and a gray, flocculent precipitate is formed.

(2) Determine the infrared absorption spectra of Prednisolone Acetate, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum in a range between 4000 cm^{-1} and 650 cm^{-1} with the Infrared Reference Spectrum or the spectrum of previously dried Prednisolone Acetate Reference Standard: both spectra exhibit similar intensities of absorption at the same wave numbers. If any difference appears, dissolve the sample and the Reference Standard in ethanol (99.5), respectively, evaporate to dryness, and repeat the test on the residues.

Optical rotation $[\alpha]_D^{20}$: +128 – +137° (after drying, 0.07

g, methanol, 20 mL, 100 mm).

Purity Other steroids—Dissolve 0.20 g of Prednisolone Acetate in exactly 10 mL of a mixture of chloroform and methanol (9:1), and use this solution as the sample solution. Separately, dissolve 0.020 g each of prednisolone, cortisone acetate and hydrocortisone acetate in exactly 10 mL of a mixture of chloroform and methanol (9:1). Pipet 1 mL of this solution, add a mixture of chloroform and methanol (9:1) to make exactly 10 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5 μL each of the sample solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of dichloromethane, diethyl ether, methanol and water (385:75:40:6) to a distance of about 15 cm, and air-dry the plate. Examine under ultraviolet light (wavelength: 254 mm): the spots from the sample solution corresponding to those from the standard solution are not more intense than the spots from the standard solution, and any spot from the sample solution other than the principal spot and the spots from prednisolone, cortisone acetate and hydrocortisone acetate does not appear.

Loss on drying Not more than 1.0% (0.5 g, 105°C, 3 hours).

Residue on ignition Not more than 0.1% (0.5 g).

Assay Dissolve about 0.01 g each of Prednisolone Acetate and Prednisolone Acetate Reference Standard, previously dried and accurately weighed, in 60 mL each of methanol, add exactly 2 mL each of the internal standard solution, then add methanol to make 100 mL, and use these solutions as the sample solution and the standard solution. Perform the test with 10 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the ratios, Q_T and Q_S, of the peak height of prednisolone acetate to that of the internal standard.

$$\text{Amount (mg) of } C_{23}H_{30}O_6 = \text{amount (mg) of Prednisolone Acetate Reference Standard} \times \frac{Q_T}{Q_S}$$

Internal standard solution—A solution of butyl parahydroxybenzoate in methanol (3 in 1000).

Operating conditions—

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

Column: A stainless steel column about 4 mm in inside diameter and about 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 25°C.

Mobile phase: A mixture of water and acetonitrile (3:2).

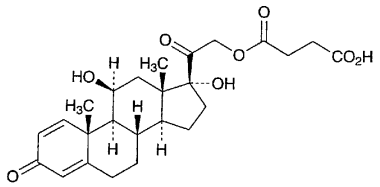
Flow rate: Adjust the flow rate so that the retention time of prednisolone acetate is about 10 minutes.

Selection of column: Proceed with 10 μL of the standard solution under the above operating conditions, and calculate the resolution. Use a column giving elution of prednisolone acetate and the internal standard in this order with the resolution between these peaks being not less than 10.

Containers and storage Containers—Tight containers.

Prednisolone Succinate

コハク酸プレドニゾロン



$C_{25}H_{32}O_8$: 460.52

11β,17,21-Trihydroxypregna-1,4-diene-3,20-dione 21-(hydrogen succinate) [*2920-86-7*]

Prednisolone Succinate, when dried, contains not less than 97.0% and not more than 103.0% of $C_{25}H_{32}O_8$.

Description Prednisolone Succinate occurs as a white, fine, crystalline powder. It is odorless.

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

Melting point: about 205°C (with decomposition).

Identification **(1)** To 2 mg of Prednisolone Succinate add 2 mL of sulfuric acid, and allow to stand for 2 to 3 minutes: a deep red color, without fluorescence, develops. To this solution add 10 mL of water cautiously: the color disappears and a gray, flocculent precipitate is formed.

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

Optical rotation $[\alpha]_D^{20}$: +114 – +120° (after drying, 0.067 g, methanol, 10 mL, 100 mm).

Purity Other steroids—Dissolve 0.10 g of Prednisolone Succinate in methanol to make exactly 10 mL, and use this solution as the sample solution. Separately, dissolve 0.030 g of prednisolone in methanol to make exactly 10 mL. Pipet 1 mL of the solution, add methanol to make exactly 10 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 5 μL of the sample solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of ethyl acetate and ethanol (95) (2:1) to a distance of about 10 cm, and air-dry the plate. Examine the plate under ultraviolet light (main wavelength: 254 nm): the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

Loss on drying Not more than 0.5% (1 g, in vacuum, phosphorus (V) oxide, 60°C, 6 hours).

Residue on ignition Not more than 0.10% (1 g).

Assay Weigh accurately about 0.01 g each of Prednisolone Succinate and Prednisolone Succinate Reference Standard,