

Hydrochloride, previously dried, dissolve in 5 mL of formic acid, add 60 mL of acetic anhydride, and titrate with 0.1 mol/L perchloric acid VS (potentiometric titration). Perform a blank determination, and make any necessary correction.

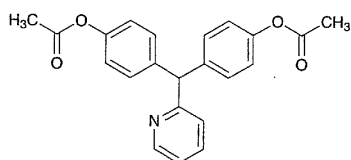
Each mL of 0.1 mol/L perchloric acid VS
= 34.793 mg of $C_{21}H_{29}NO.HCl$

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Bisacodyl

ビスコジル



$C_{22}H_{19}NO_4$: 361.39

4,4'-(Pyridin-2-ylmethylene)bis(phenyl acetate) [603-50-9]

Bisacodyl, when dried, contains not less than 98.5% of $C_{22}H_{19}NO_4$.

Description Bisacodyl occurs as a white, crystalline powder.

It is freely soluble in acetic acid (100), soluble in acetone, slightly soluble in ethanol (95) and in diethyl ether, and practically insoluble in water.

It dissolves in dilute hydrochloric acid.

Identification (1) Determine the absorption spectrum of a solution of Bisacodyl in ethanol (95) (3 in 100,000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of a solution of Bisacodyl Reference Standard prepared in the same manner as the sample solution: both spectra exhibit similar intensities of absorption at the same wavelengths.

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

Melting point 132 – 136°C

Purity (1) Chloride—Dissolve 1.0 g of Bisacodyl in 30 mL of acetone, and add 6 mL of dilute nitric acid and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution as follows: to 0.35 mL of 0.01 mol/L hydrochloric acid VS add 30 mL of acetone, 6 mL of dilute nitric acid and water to make 50 mL (not more than 0.012%).

(2) **Sulfate**—Dissolve 1.0 g of Bisacodyl in 2 mL of dilute hydrochloric acid, and add water to make 50 mL. Per-

form the test using this solution as the test solution. Prepare the control solution as follows: to 0.35 mL of 0.005 mol/L sulfuric acid VS add 2 mL of dilute hydrochloric acid and water to make 50 mL (not more than 0.017%).

(3) **Heavy metals**—Proceed with 2.0 g of Bisacodyl according to Method 4, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

(4) **Related substances**—Dissolve 0.20 g of Bisacodyl in 10 mL of acetone, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add acetone to make exactly 200 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μ L 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 2-butanone, chloroform and xylene (1:1:1) to a distance of about 10 cm, and air-dry the plate. Examine 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, 105°C, 2 hours).

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

Assay Weigh accurately about 0.5 g of Bisacodyl, previously dried, dissolve in 50 mL of acetic acid (100), and titrate with 0.1 mol/L perchloric acid VS until the color of the solution changes from orange-yellow to green (indicator: 0.5 mL of *p*-naphtholbenzein TS). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 36.140 mg of $C_{22}H_{19}NO_4$

Containers and storage Containers—Well-closed containers.

Bisacodyl Suppositories

ビスコジル坐剤

Bisacodyl Suppositories contain not less than 90% and not more than 110% of the labeled amount of bisacodyl ($C_{22}H_{19}NO_4$: 361.39).

Method of preparation Prepare as directed under Suppositories, with Bisacodyl.

Identification (1) To a quantity of Bisacodyl Suppositories, equivalent to 6 mg of Bisacodyl according to the labeled amount, add 20 mL of ethanol (95), warm on a water bath for 10 minutes, shake vigorously for 10 minutes, and allow to stand in ice water for 1 hour. Centrifuge the solution, filter the supernatant liquid, and to 2 mL of the filtrate add ethanol (95) to make 20 mL. Determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry: it exhibits a maximum between 261 nm and 265 nm.

(2) Use the filtrate obtained in (1) as the sample solution. Separately, dissolve 6 mg of Bisacodyl Reference Standard in 20 mL of ethanol (95), and use this solution as the standard solution. Perform the test with these solutions as