Yokukansankachimpihange Extract

抑肝散加陳皮半夏エキス

Change the origin/limits of content as follows:

Yokukansankachimpihange Extract contains not less than 0.6 mg and not more than 2.4 mg of saikosaponin b_2, not less than 10 mg and not more than

30 mg of glycyrrhizic acid ($C_{42}H_{62}O_{16}$: 822.93), not less than 18 mg and not more than 72 mg of hesperidin, and not less than 0.15 mg of total alkaloids (rhynchophylline and hirsutine), per extract prepared with the amount specified in the Method of preparation.

Add the following next to the Assay (3):

Assay

(4) Total alkaloids (rhyncophylline and hirsutine)—Weigh accurately about 1 g of the dry extract (or an amount of the viscous extract, equivalent to about 1 g of the dried substance), add 20 mL of diethyl ether, shake, then add 3 mL of 1 mol/L hydrochloric acid TS and 7 mL of water, and shake for 10 minutes, centrifuge, and remove the diethyl ether layer. To the aqueous layer add 20 mL of diethyl ether, and proceed in the same manner as above. To the aqueous layer add 10 mL of sodium hydroxide TS and 20 mL of diethyl ether, shake for 10 minutes, centrifuge, and separate the diethyl ether layer. To the aqueous layer add 20 mL of diethyl ether, proceed in the same manner, and repeat this procedure twice. Combine all the extracts, evaporate the solvent under low pressure (in vacuo) at not more than 40°C, dissolve the residue in the mobile phase to make exactly 10 mL, and use this solution as the sample solution. Separately, weigh accurately about 5 mg each of rhyncophylline for assay and hirsutine for assay, and dissolve in a mixture of methanol and dilute acetic acid (7:3) to make exactly 100 mL. Pipet 10 mL of this solution, add the mixture of methanol and dilute acetic acid (7:3) to make exactly 50 mL, and use this solution as the standard solution. Perform the test with exactly 10 μL each of the sample solution and standard solution as directed under Liquid Chromatography <*2.01*> according to the following conditions, and determine the peak areas, A_{TR} and A_{TH}, and A_{SR} and A_{SH}, of rhyncophylline and hirsutine, in each solution.

Amount (mg) of total alkaloids (rhyncophylline and hirsutine)

$$= (M_{SR} \times A_{TR}/A_{SR} + M_{SH} \times A_{TH}/A_{SH}) \times 1/50$$

M_{SR}: Amount (mg) of rhynchophylline for assay taken

M_{SH}: Amount (mg) of hirsutine for assay taken

Operation conditions—

Detector: An ultraviolet absorption photometer (wavelength: 245 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 (5 μm in particle diameter).

Column temperature: A constant temperature of about 40°C.

Mobile phase: To 1 g of sodium lauryl sulfate add 600 mL of methanol, shake, then add 400 mL of water and 5 mL of acetic acid (100) to dissolve sodium lauryl sulfate.

Flow rate: 1.0 mL per minute.

Systemic suitability—

System performance: When the procedure is run with 10 μL of the standard solution under the above operating conditions, the number of theoretical plates and the symmetry factor of the peaks of rhyncophylline and hirsutine are not less than 5000 and not more than 1.5, respectively.

System repeatability: When the test is repeated 6 times with 10 μL of the standard solution under the above operating conditions, the relative standard deviations of the peak area of rhyncophylline and hirsutine is not more than 1.5%, respectively.