陕西绿清生物工程有限公司

Shaanxi Green Bio-Engineering Co.,Ltd

Shilaji Extract Test Method

The FA in the sample is extracted with acid. In a strong acid medium, the FA is oxidized with excess potassium dichromate, and the remaining potassium dichromate is titrated with standard ferrous ammonium sulfate solution.

Ritual and design

Laboratory instruments and equipment commonly used

Reagents and solutions

(1) Potassium dichromate standard solution: 0.1 mol/L, weigh 4.9036 grams of working reference reagent potassium dichromate, dissolve it in 1 liter of distilled water, and store it in a narrow-necked bottle for later use.

(2) Potassium dichromate (GB642) solution: 0.8mol/L, weigh 40g analytically pure potassium dichromate, dissolve in 1 liter of water, and store in a narrow mouth for later use.

(3) Sulfuric acid (GB 625). The specific gravity is 1. 84g/cm³.

(4) o-phenanthroline (GB1293) indicator: weigh 1.5 g of o-phenanthroline and 1 g of ferrous ammonium sulfate and dissolve in 100ml for distillation

Water, store in a brown bottle for preservation.

(5) Standard solution of ferrous ammonium sulfate: c[Fe*]=0.1 mol/L.

Measurement procedure

Accurately weigh 0.2-0.5g of the sample (depending on the content, accurate to 0.0002g), add 0.1% H2SO4 to a 250mL Erlenmeyer flask, the solution is 70mL, insert a small glass funnel into the bottle and place it in the water at 45-100℃ and heat for 0.5~1h , Shake the Erlenmeyer flask intermittently, take it out and cool it and filter it into a 250mL volumetric flask, wash the residue until the solution is colorless, and finally make the volume up to the mark (0.1% H2SO4 for extraction and 0.1% sulfuric acid to make the volume at the same time).

Take 5mL of the above solution, (if FA content is less than 10%, take 10mL), in a 250mL Erlenmeyer flask, add 0.8mol/L potassium dichromate 5mL, concentrated, H2SO4, 15mL, boil on a boiling water bath for 0.5h, Cool to room temperature, add 50-60mL distilled water, 2-4 drops of o-phenanthroline indicator, and titrate with 0.1mol/L standard ferrous ammonium sulfate solution

to change from orange-red to green and then to wine-red as the end point. At the same time, make a blank test.

Calculate result

$$FA\%=\frac{(VO-V)XMX0.003}{GXC}X\frac{250}{5}（或\frac{250}{10}）X100\%$$

V. --The standard solution of ferrous ammonium sulfate consumed in titrating the blank, ml;

V-a standard solution of ferrous ammonium sulfate consumed in a titration sample, ml;

M--concentration of standard ferrous ammonium sulfate solution, mol/L;

G-the weight of the sample, g;

The carbon coefficient of C-FA can be calculated as 0.48-0.50. Before unification, it is recommended to measure it based on your own raw materials.