山东泽胜化工有限公司

SHANDONG ZESHENG CHEMICAL CO., LTD.

MOA OF 2-Deoxy-D-glucose

STANDARD NAME: 2-Deoxy-D-glucose

APPEARANCE: White or lightly yellow powder

BATCH NO.:ZESH-210218

[Assay determination]

Reagent And Solution

1.Sulfuric acid solution[c(½H2SO4)]=0.01mol/L

2.Water:Primary water

3.Sulfuric acid solution 2-deoxy-D-glucose series standard solution: weigh 2-deoxy-D-glucose dried at 105 ℃ ± 2 ℃ to constant weight, and prepare 5 series standard solutions with different concentrations ranging from 0.1mg/ml to 10mg / ml with sulfuric acid solution.

Instruments And Equipment

1. High performance liquid chromatography (with differential refraction

2. detector and column constant temperature system)

2.Mobile phase vacuum filter degassing device and 0.2 μ 45 m or 0 μ M micro porous membrane

3.Analytical balance: accuracy 0.1mg

4.Micro injector: 50 μ L

Reference chromatographic conditions

1.Chromatographic column: aminex hpx-8h. Or equivalent analytical column.

2.Mobile phase: sulfuric acid solution

3.Detector temperature: 40 ℃

4.Column temperature: 45 ℃

5.Flow rate: 0.7ml/min

6.Injection volume: 20 μ L.

Analysis steps

1.Preparation of sample solution

Weigh about 0.1g of sample (accurate to 0.0001g. based on dry basis), add 1.0ml of water solution, add sulfuric acid solution to constant volume, and control the concentration within the concentration range of standard curve, and use 0.2 μ M = 0.45 μ M water phase microporous membrane filtration, filtrate for standby.

2.Draw standard curve

After injection of 2-deoxy-D-glucose series standard solutions, the peak surface was measured with the concentration of series standard solutions plot the standard curve. The linear relationship should be above 0.9990.

3.Sample determination

The standard solution of 2-deoxy-D-glucose and the prepared sample solution were injected respectively. The chromatographic peak of 2-deoxy-D-glucose was determined according to the retention time of the standard solution. According to the peak area of the sample, the content of 2-deoxy-D-glucose was calculated by external standard method.

4.Result calculation

The content of 2-deoxy-D-glucose in the sample is calculated according to formula (1), and the value is expressed in%.

$$X_1 = \frac{cV \times 10^{-3}}{m} \times 100 \qquad \cdots\cdots (1)$$

X2 -mass fraction of 2-deoxy-D-glucose in the sample (measured by dry basis),%;

C-The concentration of 2-deoxy-D-glucose in the sample solution was obtained by C-curve, in mg / ml;

V-dilution volume of sample, unit: ml;

M-mass of the sample (in dry basis), in grams (g)

Precision

Under the condition of repeatability, the absolute difference between the

two independent determination results should not exceed 0.5% of the arithmetic mean value.

【Loss on drying】Take the appropriate amount of this product and dry it to constant weight at 105 °C. The weight loss should not exceed 1.0%

【Specific Rotation】Weigh about 10 g (accurate to 0.0001 g) of the sample, put it into a 100 ml volumetric flask, add appropriate amount of water to dissolve it, add 0.2 ml of ammonia to dissolve it, use water to fix the volume to the scale, shake it well, place it for 10 min, adjust it to zero at 20 ℃ ± 0.5 ℃. Then rinse the polarizing tube twice with the sample solution. The sample solution is filled with the polarizing tube, and no bubbles can be produced. The solution of liquid or solid substance for determination shall not be turbid or contain suspended particles. In case of the above situation, filter in advance and discard the primary filtrate.

The specific rotation of the sample is calculated according to formula (2)

$$X_2 = \frac{a \times 100}{m \times L \times (1 - X_0)} \qquad \cdots\cdots (2)$$

X2 ——specific rotation, in degrees (°)

a ——optical rotation, in degrees (°)

100 ——volume of sample solution, unit: ML

M——mass of sample, in gram (g)

L——length of the polarizer, in Decimeter (DM)

X0 ——mass fraction of water in the sample (%)

【Heavy Metals】Take an appropriate amount of this product, dissolve it with 23ml water, add 2ml acetic acid buffer solution (pH3.5), check it according to the specified method, and compare it with the standard lead solution (every 1ml is equal to 10μgPb 2.0ml,which should not be deeper than the control solution prepared by the same method.

【Identification】HPLC

【Individual impurity】HPLC

【Total impurity】HPLC

【Residue on ignition】The crucible was heated and washed with hydrochloric acid, then washed with white water, and then rinsed with distilled water. Place the cleaned crucible in a high temperature furnace, burn it for 0.5 h at 525 ℃ ± 25 ℃, take it out and cool it to below 200 c at room temperature, put it into a dryer to cool it to room temperature, accurately weigh it, and repeat the burning point to constant weight (the difference between the two weighing is not more than 0.5 mg).

Weigh about 2 g (accurate to 0.000 1 g) of the sample, put it into a crucible which has been burned to a certain weight, add 1 ml of concentrated sulfuric acid, rotate it slowly to make it uniform, and heat it

carefully on the electric furnace until it is completely carbonized. Then, put it into a high-temperature furnace and burn it at 525 C and 25 C. keep the temperature until all the carbonization disappears (at least 2 h). Take it out for cooling, add a few drops of concentrated sulfuric acid to wet the residue, put it into the high-temperature protection again for burning, and point it to complete ashing. Take it out, cool it to below 200 ℃ at room temperature, put it into a dryer, cool it to room temperature, accurately weigh it, and repeatedly burn it until constant weight (the difference between the two weighing is not more than 0.3mg).