

6 conductance ash

6.1 the principle

The conductivity reflects the concentration of ionized water-soluble salts. The conductivity of the known sugar solution is measured, and then the conductivity ash can be calculated by using the conversion coefficient. The concentration of the sugar solution used in this method is 31.3 g/100 ml.

6.2 Instruments and equipment

Conductivity meter.

6.3 reagent

Unless otherwise stated, the reagents used in this method are analytically pure and the water is tertiary water specified in GB/T 6682.

6.3.1

Distilled water or DEionized water: Refined white granulated sugar is applied to heavy distilled water (distilled twice) or deionized water with a conductivity of less than 2 $\mu\text{S}/\text{cm}$. For other grades of granulated sugar, distilled water with conductivity less than 15 $\mu\text{S}/\text{cm}$ is allowed.

6.3.2

0.01mol /L potassium chloride solution: Pure grade potassium chloride was taken, heated to 500 $^{\circ}\text{C}$, dehydrated for 30 min, cooled, weighed 0.7455g, dissolved in 1000 mL volume flask, and added water to the line.

6.3.3

0.0025 mol/L potassium chloride solution: Absorb 50 mL of 0.01 mol/L potassium chloride solution into 200 mL volumetric flask, add water and dilute to the line. The conductivity of the solution at 20 $^{\circ}\text{C}$ is 328 $\mu\text{S}/\text{cm}$.

Step 6.4

6.4.1 determination

Said take sugar 31.3 g \pm 0.1g in the dry clean beaker, add distilled water dissolved and out into a 100 mL volumetric flask and the beaker is washed with distilled water for many times and the glass rod, wash water into the volumetric flask, along with all the add distilled water to the line, shake well, with the determination of liquid washing first conductivity with the conductivity electrode and dry clean small beaker 2 ~ 3 times, and then into the sample solution, sample liquid conductivity is measured by conductivity meter, record reading and reading at the time of the sample liquid temperature.

The conductance pool constant was checked and measured by 0.0025 mol/L potassium chloride solution.

6.4.2 Calculation and result representation

The conductance ash content C of the white granulated sugar sample is calculated according to Equation (9) and expressed as mass fraction.

$$C = 6 \times 10^{-4} \times (C_1 - 0.35C_2)$$

C - conductance ash in grams per 100 grams (g/100 g);

C₁-31.3 the conductivity of g/100 mL sugar solution at 20.0 $^{\circ}\text{C}$, in microcysts per centimeter ($\mu\text{S}/\text{cm}$);

C₂- Electrical conductivity of distilled water used for sugar dissolution at 20.0 °C, in microsieverts per centimeter (μS/cm),

6.4.3 Temperature correction

The standard temperature for conductivity measurement is 20.0 °C. If it is not 20.0 °C, it will be corrected according to Formula (10), but the measured temperature should not be exceeded generally 20.0 °C ±5.0 °C. Temperature correction for electrical conductivity of distilled water for dissolved sugars was negligible as the effect was minimal.

$$C_{20.0}=C_t / (1+0.026 (t-20))$$

Type:

C_{20.0}-- the conductivity of the sugar solution at 20.0°C, in microsieverts per centimeter (μS/cm);

C_t. The electrical conductivity of the sugar solution at a temperature of t°C, in microsieverts per centimeter (μS /cm);

t: The temperature of the sugar liquid in Celsius (°C) for the determination of its electrical conductivity.

Represented by the arithmetic mean of two independent measurements obtained under repetitive conditions, with two significant digits retained.

5.1 Sample Preparation Note: Sample contamination should be avoided during sampling and sample preparation.

5.1.1 Grain and bean samples shall be crushed and stored in plastic bottles after the sundries are removed.

5.1.2 Wash vegetables, fruits, fish, meat and other samples with water, dry them, take edible parts, make homogenate, and store them in plastic bottles.

5.1.3 Shake well the samples of beverage, wine, vinegar, soy sauce, edible vegetable oil, liquid milk and other liquid samples.

5.2 Sample pretreatment

5.2.1 The solid sample was digested by wet digestion, 0.2g~3g(accurate to 0.001g) was weighed, or the liquid sample was transferred into the digestive tube with scale, 10mL nitric acid and 0.5mL perchloric acid were added, and then digested in an adjustable electric heating furnace (reference condition :120 °C/0.5h~1h; Rise to 180 °C/2h~4h, 200 °C~220 °C). If the digestive juice is brown, add a small amount of nitric acid to dissolve it until it is white smoke, and the digestive juice is colorless, transparent or slightly yellow. Take out the digestive tube, cool it down with water for a constant volume of 10mL, and mix it well for later use. At the same time do reagent blank test. A conical flask on an adjustable electric heating plate can also be used for wet digestion according to the above operation method

5.2.2 Microwave digestion Solid sample 0.2g~0.8g(accurate to 0.001g) or liquid sample 0.500ml ~ 3.00ml was accurately transferred into the microwave digestion tank, and 5mL nitric acid was added. The sample was then dissolved according to the operation steps of microwave digestion. Refer to Appendix A for the digestion conditions. After cooling, take out the digestion tank and pour acid on the electric heating plate at 140 °C~160°C to about 1mL. After the digestion tank is cooled, the digestion solution is transferred to a 10mL volumetric bottle, and a small amount of water is used to wash the digestion tank for 2 ~3 times. The washing solution is combined in the

volumetric bottle, and the washing solution is adjusted to the scale with water, and then mixed for later use. At the same time do reagent blank test.

5.2.3 Pressure tank digestion

The solid sample was weighed 0.2g~1g(accurate to 0.001g) or the liquid sample was transferred 0.500ml ~ 5.00ml accurately into the digestion tank and 5mL nitric acid was added. Cover the inner cover, tighten the stainless steel coat, put it into a constant temperature drying oven, keep it at 140 °C~160 °C for 4h~5h. After cooling, loosen the outer tank slowly, take out the digestion inner tank, and put it on an adjustable electric heating plate to drive the acid to about 1mL at 140 °C~160 °C. After cooling, transfer the digestion solution to a 10mL volumetric bottle, wash the inner tank and inner cover with a small amount of water for 2 ~3 times, and combine the washing solution in the volumetric bottle with water to the scale, and mix it for later use. At the same time do reagent blank test.

5.3 the determination of

5.3.1 Instrument reference conditions Shall be adjusted to the optimal state according to the performance of each instrument. See Appendix B for reference terms.

5.3.2 standard curve produced by the mass concentration of 10 µg/L, respectively, from low to high order will L lead standard series of solution and ammonium dihydrogen phosphate - 5 µg/L palladium nitrate solution (can be according to the use of the instrument to determine the best sampling amount) into the graphite furnace at the same time, measuring the absorbance values after atomization, mass concentration as the abscissa, absorbance value as the ordinate, making standard curve.

5.3.3 Determination of Sample Solution Under the same experimental conditions as the standard solution, 10 µg/L blank solution or sample solution and 5 µg/L ammonium dihydrogen phosphate - palladium nitrate solution (the optimal amount of sample injection can be determined according to the instrument used) were injected into the graphite furnace at the same time. After atomization, the absorbance was measured to compare quantitative with the standard series.

6. Presentation of analysis results

The lead content in the sample is calculated according to Formula (1):

$$X=(P-P_0) \times V / (m \times 1000)$$

X-- the amount of lead in the sample in milligrams per kilogram or milligrams per liter (mg/kg or mg/L);

P-- the mass concentration of lead in a sample solution in micrograms per liter (µg/L);

P₀-- the mass concentration of lead in blank solution in micrograms per liter (µg/L);

V -- constant volume of the sample digestion solution, in mL;

M -- The sample weight or transfer volume, in grams or milliliters (g or mL);

1000 -- conversion factor.

When the lead content is ≥ 1.00mg /kg(or mg/L), three significant digits are retained in the calculation results; When the lead content is less than 1.00 mg/kg(or mg/L), the results retain

two significant digits.

7 precision

The absolute difference between the two independent measurements obtained under repeatability conditions must not exceed 20% of the arithmetic mean.

8. Other

When the sample weight was 0.5g(or 0.5mL) and the volume was 10mL, the detection limit of the method was 0.02mg/kg(or 0.02mg/L) and the determination limit was 0.04mg/kg(or 0.04mg/L).