Attachment: 4201 Determination of Hydrolytic Resistance for Glass Grains at 121℃

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The hydrolytic resistance of glass grains at 121□ can evaluate the resistance of glass material to water erosion. The test is that a certain amount of glass grains with specified size is corroded by the prescribed amount of water in the specified apparatus，and under specified conditions. The hydrolytic resistance is determined by titration of the extraction solution.

Instruments: Autoclave, electronic balance, burettes, conical flasks, beakers (Note: The ~~glass container~~ conical flasks and beakers must be made of borosilicate glass or quartz glass. The mean linear thermal expansion coefficient is about $3.3\times10^{-6}K^{-1}$ for borosilicate glass. New glass containers must undergo repeated aging treatment according to the specified procedures until the water is neutral to 0.025% methyl red sodium aqueous solution. Oven, hammer, mortar, and pestle made of hardened-steel (see Figure). Permanent magnet, a set of stainless steel sieves (containing sieve A: 425 μm; sieve B: 300 μm; sieve O: 600-1000 μm).

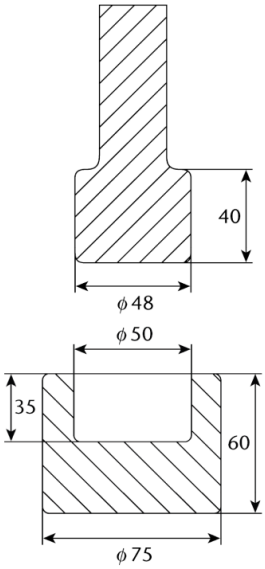

Figure Mortar and pestle (dimensions in millimeters)

The test water should comply with the following requirements:

(1) The conductivity of the test water shall not exceed 0.1 mS/m at 25□±1□.

(2) To remove carbon dioxide and other dissolved gas, the test water should be boiled vigorously for more than 15 min in an aging treated conical flask.

(3) The test water shall be neutral to the 0.025% methyl red sodium aqueous solution. The color of the water turns to orange-red (pH5.4-5.6) after adding 4 drops of 0.025% sodium methyl red sodium solution to 50 ml of water. The test water can be used for blank test.

Sample preparation: Crush the sample into pieces. Place some broken pieces

into the mortar, insert the pestle, and strike it heavily with the hammer once only. Transfer the samples in the mortar to the coarsest sieve O of the set. Repeat the above operation process. Shake the set of sieves for 5 min with a mechanical sieve-shaker (or manually shake the set of sieves). Transfer the glass grains that pass through sieve A and stay on sieve B to a weighing bottle. The glass grains shall be more than 10 g and prepare three samples in total.

Remove iron particles with the permanent magnet. Transfer the glass grains into a 250 ml conical flask. Add 30 ml of anhydrous ethanol or acetone to wash the glass grains for at least 6 times until the acetone or anhydrous ethanol remains clear. Decant the anhydrous ethanol or acetone in conical flask completely after each wash. Dry the glass grains by putting the conical flask on a warm plate or other heating device to remove the residual anhydrous ethanol or acetone, then heat at 140□ for 20 min in a drying oven. Transfer the conical flask with dried glass grains into a desiccator to cool. The storage time shall not be more than 24 hours.

Determination: Accurately weigh 10 g of glass grains described above into a 250 ml conical flask and accurately add 50 ml of test water. Cap the flasks with inverted beakers so that the inner bottom of the beakers fit snugly down onto the top rims of the flasks. The flasks also can be covered with other suitable materials. Place all the flasks into the autoclave. Leave the vent-cock open and heat the autoclave at a regular rate. The steam is released vigorously from the vent-cock after 20-30 min and maintain a vigorous release for 10 min. Close the vent-cock, and raise the temperature to 121□±1□ at a rate of 1□/min within 20-22 min, counting from the time the when this temperature is reached. Maintain the temperature at 121□±1□ for 30min±1min. Cool down and decompress slowly to 100□ within 40-44min (to prevent the formation of vacuum). Take out the samples when the temperature is lower than 95□, and cool them to room temperature. Take the test water for a blank, carry out the ~~test~~ same procedures as the test solution. The results of the titration are corrected by the blank test. The titration must be completed within 1 hour after removing from the autoclave.

Add 4 drops of 0.025% methyl red sodium aqueous solution to each of the flasks. Titrate the test solution immediately with hydrochloric acid (0.02 mol/L) VS until the color matches that obtained with the blank solution.

Result representation: Calculate the mean value of the consumed volume in milliliter of hydrochloric acid (0.02 mol/L) VS per g of the glass grains.

Repeat the test if the highest and lowest values differ by more than the permissible range given in Table 1.

Table 1 Permissible range for values obtained

Mean of the values obtained for the consumption of hydrochloric acid (0.02mol/L) VS per g of glass grains (ml/g)	Permissible range of the values obtained
≤0.10	25% of the mean
>0.10~0.20	20% of the mean
>0.20	10% of the mean

Grade: The hydrolytic resistance of glass grains should be classified according to the consumed volume in milliliter of hydrochloric acid (0.02mol/L) VS in Table 2.

Table 2 Grade of hydrolytic resistance of glass grains

Grade	Volume of hydrochloric acid (0.02mol/L) VS per g of test glass grains (ml)
1	≤0.10
2	>0.10~0.85

Drafted by: National Institutes for Food and Drug Control Contact number: 010-67095110