Attachment: 4203 Determination of Boron Trioxide Content for Glass

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Boron trioxide is one of the key components of borosilicate glass, and its content can be used for the characterization of borosilicate glass materials and formula stability

This method applies to the determination of boron trioxide content in borosilicate glass for pharmaceutical use.

Determination Principle: The glass is pulverized and ground to powder. After the powered glass melted by alkali and react with acid, calcium carbonate is used to make boron form calcium borate, which is easily soluble in water, therefore separate from other elements. Mannitol is added to make boric acid quantitatively converted to alcohol boric acid, which is then titrated with sodium hydroxide. The amount of boron trioxide contained in the glass sample is calculated from the concentration and consumed volume of sodium hydroxide (VS).

Sample Preparation: Take an appropriate amount of cleaned samples. Crush and grind the parts without printing to fine powder (the particle size shall be less than 100 μm). Dry the fine glass powder under 105 to 110℃ for at least 1 hour, and cool it in a desiccator for 1 hour or more for later use. The prepared samples need to be re-dried if stored in the desiccator for more than 24 hours.

Determination Take about 0.5 g of the prescribed fine glass powder, accurately weighed. Put the sample into a platinum crucible, add 4 g of anhydrous sodium carbonate. Rotate the crucible slowly to make sure that the sample are fully mixed with the anhydrous sodium carbonate. Cover the crucible and melt the sample with a flame blowtorch for 5 to 15 minutes, or melt at 850 to 900℃ for 15 to 30 minutes (Or use other appropriate heating methods until the sample is completely melted). Or add 4 g of sodium hydroxide into a nickel or a silver crucible, heat until the sodium hydroxide is melted, and allow to cool down. Take about 0.5 g of the prescribed fine powered glass, accurately weighed and place it into the crucible. Cover the crucible, and melt the sample with a flame blowtorch for 5 to 15 minutes, or melt the sample under 400 to 450 ℃ for about 15 to 30 minutes (Or use other appropriate heating methods until the sample were completely melted). Allow the sample to cool down in the air (note: pay attention to prevent the sample from volatilizing during the melting process; the melting times for different kinds of glass are slightly different).

Leach the residue with a small amount of hot water and transfer it to a tall beaker, and add 20 ml of hydrochloric acid to disperse the residue. Then wash the crucible and the cover for several times with no more than 5 ml of hydrochloric acid solution (1→2) in total, combine the rinsing in the beaker. After the residue is dissolved completely, neutralize the remaining acid with calcium carbonate. Add about 4 g of excess calcium carbonate, boil the beaker in water bath for about 30 minutes, then filter with fast filter paper while hot, and wash the beaker and precipitate with hot water for several times, add a small amount (about 0.3 to 0.5 g) of EDTA disodium to the filtrate and bring to boil.

Remove the beaker from the heater and cool it to room temperature. Add 2 drops of 0.1% methyl red ethanol solution. Adjust the solution to neutral (in bright yellow) with 0.l mol/L sodium hydroxide solution and 0.1 mol/L hydrochloric acid. Add l ml of 0.1% phenolphthalein ethanol indicator and 2 to 3 g of mannitol (acidic color indicated by methyl red), titrate with sodium hydroxide (VS) (0.l mol/L) to a reddish color (basic color indicated by phenolphthalein). Then add about l g of mannitol again, shake gently, if the reddish color fades away, titrate with sodium hydroxide (VS) (0.l mol/L) to a reddish color, repeat the procedure until the reddish color does not fade after adding mannitol. Read the volume of the sodium hydroxide (VS) (0.l mol/L) consumed.

Take a crucible made of the same material to perform the blank test, and apply the blank

test to correct the titration results. Per 1 ml of sodium hydroxide (VS) (0.l mol/L) is equivalent to 3.481 mg of boron trioxide.

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