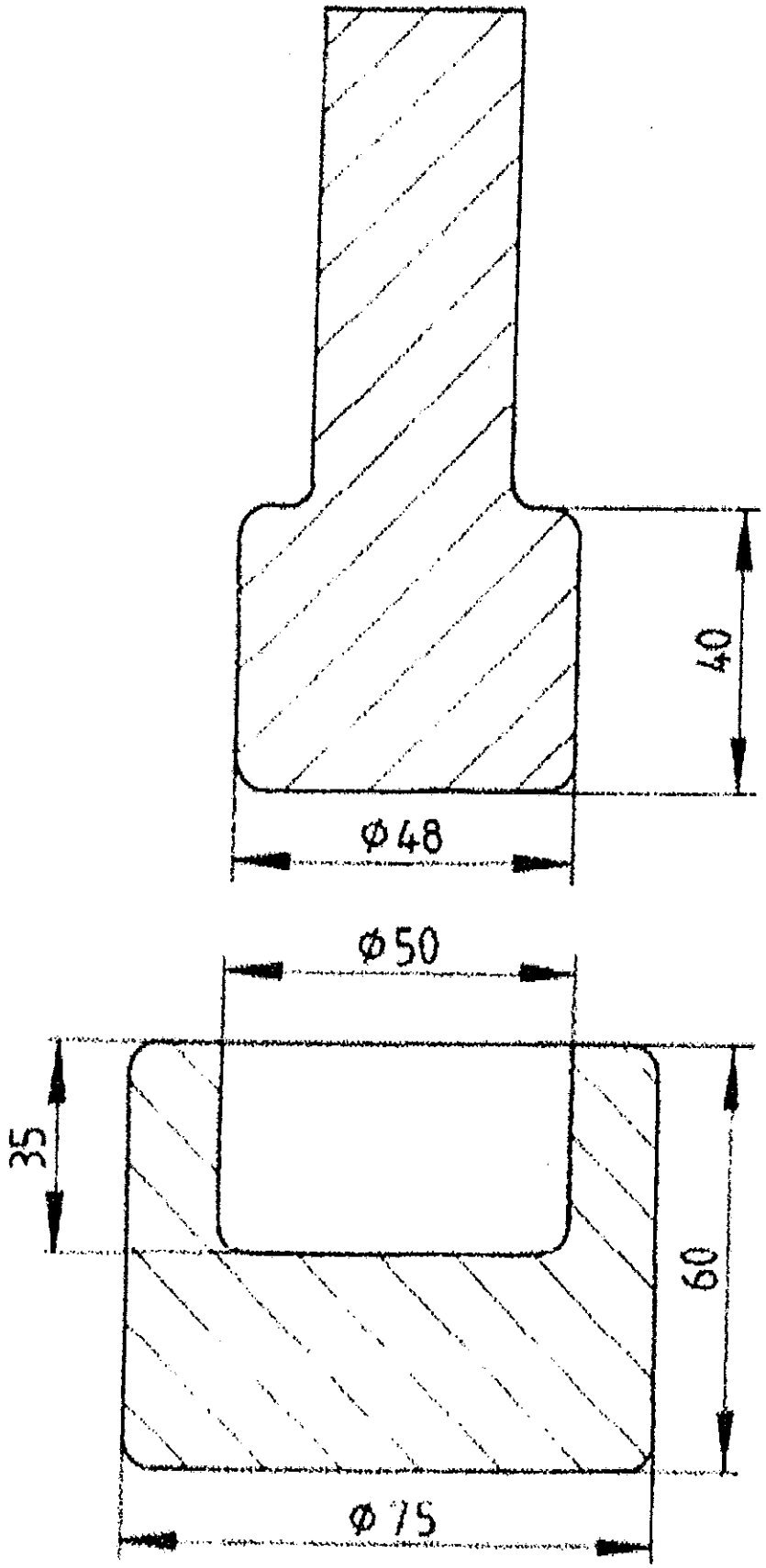


Figure 1. Mortar and Pestle for Pulverizing Glass[1]

*Other Equipment*— Also required are 20.3-cm (8-inch) sieves made of stainless steel,

including the Nos. 20, 40, and 50 sieves, along with the pan and cover (see *Sizes of Standard Sieve Series in Range of Interest* under *Particle Size Distribution Estimation by Analytical Sieving* 〈 786 〉); 250-mL conical flasks made of resistant glass aged as specified; a 900-g (2-lb) hammer; a permanent magnet; a desiccator; and an adequate volumetric apparatus.

**Reagents**—

*High-Purity Water*— The water used in these tests has a conductivity at 25°, as measured in an in-line cell just prior to dispensing, of not greater than 0.15 µS per cm (6.67 Megohm-cm). There must also be an assurance that this water is not contaminated by copper or its products (e.g., copper pipes, stills, or receivers). The water may be prepared by passing distilled water through a deionizer cartridge packed with a mixed bed of nuclear-grade resin, then through a cellulose ester membrane having openings not exceeding 0.45 µm.[2] Do not use copper tubing. Flush the discharge lines before water is dispensed into test vessels. When the low conductivity specification can no longer be met, replace the deionizer cartridge.

*Carbon Dioxide - Free Water*— This is *Purified Water* that has been boiled vigorously for 5 minutes or more and allowed to cool while protected from absorption of carbon dioxide from the atmosphere, or *Purified Water* that has a resistivity of not less than 18 Mohm-cm.

*Methyl Red Solution (Powdered Glass Test and Water Attack at 121°)*— Dissolve 24 mg of methyl red sodium in *Purified Water* to make 100 mL. If necessary, neutralize the solution with 0.02 N sodium hydroxide, or acidify it with 0.02 N sulfuric acid so that the titration of 100 mL of *High-Purity Water,* containing 5 drops of indicator, does not require more than 0.020 mL of 0.020 N sodium hydroxide to effect the color change of the indicator, which should occur at a pH of 5.6.

*Methyl Red Solution (Surface Glass Test)*— Dissolve 50 mg of methyl red solution in 1.86 mL of 0.1 M sodium hydroxide and 50 mL of ethanol (96%) and dilute to 100 mL with *Purified Water*. To test for sensitivity, add 100 mL of *Carbon Dioxide-Free Water* and 0.05 mL of 0.02 M hydrochloric acid to 0.1 mL of the methyl red solution (the solution should be red). Not more than 0.1 mL of 0.02 M sodium hydroxide is required to change the color to yellow. Color change: pH 4.4 (red) to pH 6.0 (yellow).

**Powdered Glass Test**

Rinse thoroughly with *Purified Water* six or more containers selected at random, and dry them with a current of clean, dry air. Crush the containers into fragments about 25 mm in size, divide about 100 g of the coarsely crushed glass into three approximately equal portions, and place one of the portions in the special mortar. With the pestle in place, crush the glass further by striking 3 or 4 blows with the hammer. Nest the sieves, and

empty the mortar into the No. 20 sieve. Repeat the operation on each of the two remaining portions of glass, emptying the mortar each time into the No. 20 sieve. Shake the sieves for a short time, then remove the glass from the Nos. 20 and 40 sieves, and again crush and sieve as before. Repeat again this crushing and sieving operation. Empty the receiving pan, reassemble the nest of sieves, and shake by mechanical means for 5 minutes or by hand for an equivalent length of time. Transfer the portion retained on the No. 50 sieve, which should weigh in excess of 10 g, to a closed container, and store in a desiccator until used for the test.

Spread the specimen on a piece of glazed paper, and pass a magnet through it to remove particles of iron that may be introduced during the crushing. Transfer the specimen to a 250-mL conical flask of resistant glass, and wash it with six 30-mL portions of acetone, swirling each time for about 30 seconds, and carefully decanting the acetone. After washing, the specimen should be free from agglomerations of glass powder, and the surface of the grains should be practically free from adhering fine particles. Dry the flask and contents for 20 minutes at 140°, transfer the grains to a weighing bottle, and cool in a desiccator. Use the test specimen within 48 hours after drying.

**Procedure**— Transfer 10.00 g of the prepared specimen, accurately weighed, to a 250-mL conical flask that has been digested (aged) previously with *High-Purity Water* in a bath at 90° for at least 24 hours or at 121° for 1 hour. Add 50.0 mL of *High-Purity Water* to this flask and to one similarly prepared to provide a blank. Cap all flasks with borosilicate glass beakers that previously have been treated as described for the flasks and that are of such size that the bottoms of the beakers fit snugly down on the top rims of the containers. Place the containers in the autoclave, and close it securely, leaving the vent cock open. Heat until steam issues vigorously from the vent cock, and continue heating for 10 minutes. Close the vent cock, and adjust the temperature to 121°, taking 19 to 23 minutes to reach the desired temperature. Hold the temperature at 121 ± 2.0° for 30 minutes, counting from the time this temperature is reached. Reduce the heat so that the autoclave cools and comes to atmospheric pressure in 38 to 46 minutes, being vented as necessary to prevent the formation of a vacuum. Cool the flask at once in running water, decant the water from the flask into a suitably cleansed vessel, and wash the residual powdered glass with four 15-mL portions of *High-Purity Water,* adding the decanted washings to the main portion. Add 5 drops of *Methyl Red Solution,* and titrate immediately with 0.020 N sulfuric acid. If the volume of titrating solution is expected to be less than 10 mL, use a microburet. Record the volume of 0.020 N sulfuric acid used to neutralize the extract from 10 g of the prepared specimen of glass, corrected for a blank. The volume does not exceed that indicated in *Table 2* for the type of glass concerned.

**Table 2. Test Limits for Powdered Glass Test**

| | | | Limits | |
|---|---|---|---|---|
| Type | General Description[a] | Type of Test | Size,[b] | mL of 0.020 N Acid |

| | | | mL | |
|---|---|---|---|---|
| I | Highly resistant, borosilicate glass | *Powdered Glass* | All | 1.0 |
| III | Soda-lime glass | *Powdered Glass* | All | 8.5 |

[a] The description applies to containers of this type of glass usually available.

[b] Size indicates the overflow capacity of the container.

**Surface Glass Test**

**Determination of the Filling Volume**— The filling volume is the volume to be filled with *Purified Water* in the container for the purpose of the test. For vials and bottles the filling volume is 90% of the brimful capacity. For ampules it is the volume up to the height of the shoulder.

**Vials and Bottle**— Select, at random, 6 containers from the sample lot, or 3 if their capacity exceeds 100 mL, and remove any dirt or debris. Weigh the empty containers with an accuracy of 0.1 g. Place the containers on a horizontal surface, and fill them with *Purified Water* to about the rim edge, avoiding overflow and introduction of air bubbles. Adjust the liquid levels to the brimful line. Weigh the filled containers to obtain the mass of the water, expressed to 2 decimal places, for containers having a nominal volume less or equal to 30 mL, and expressed to 1 decimal place for containers having a nominal volume greater than 30 mL. Calculate the mean value of the brimful capacity in mL, and multiply it by 0.9. This volume, expressed to 1 decimal place, is the filling volume for the particular container lot.

**Ampules**— Place at least 6 dry ampules on a flat, horizontal surface, and fill them with *Purified Water* from a buret until the water reaches point A, where the body of the ampule decreases to the shoulder of the ampule (see *Figure 2*). Read the capacities, expressed to 2 decimal places, and calculate the mean value. This volume, expressed to 1 decimal place, is the filling volume for the particular ampule lot. The filling volume may also be determined by weighing.

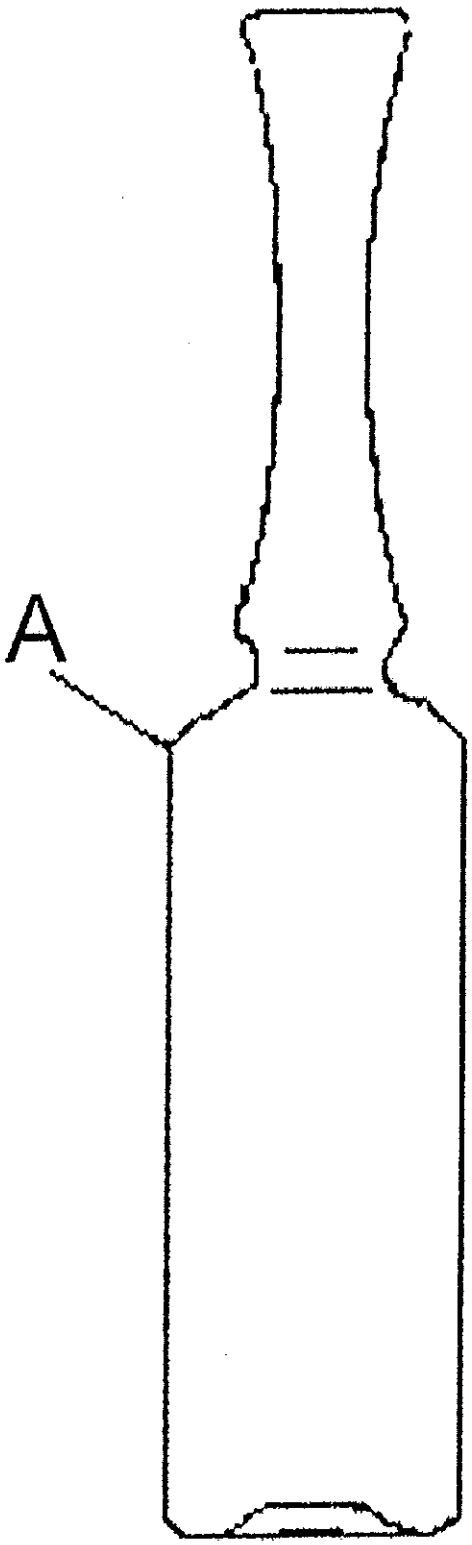

Figure 2. Filling Volumes of Ampules (up to point A)

**Test**— The determination is carried out on unused containers. The volumes of the test liquid necessary for the final determination are indicated in *Table 3*.

**Table 3. Volume of Test Liquid and Number of Titrations**

| Filling Volume (mL) | Volume of Test Liquid for One Titration (mL) | Number of Titrations |
|---|---|---|
| Up to 3 | 25.0 | 1 |
| Above 3 and up to 30 | 50.0 | 2 |
| Above 30 and up to 100 | 100.0 | 2 |
| Above 100 | 100.0 | 3 |

**Cleaning**— Remove any debris or dust. Shortly before the test, rinse each container carefully at least twice with *Purified Water*, and allow to stand. Immediately before testing, empty the containers, rinse once with *Purified Water*, then with *Carbon Dioxide-Free Water* and allow to drain. Complete the cleaning procedure from the first rinsing in not less than 20 minutes and not more than 25 minutes. Heat closed ampules in a water bath or in an air-oven at about 50° for approximately 2 minutes before opening. Do not rinse before testing.

**Filling and Heating**— The containers are filled with *Carbon Dioxide-Free Water* up to the filling volume. Containers in the form of cartridges or prefilled syringes are closed in a suitable manner with material that does not interfere with the test. Each container, including ampules, shall be loosely capped with an inert material such as a dish of neutral glass or aluminum foil previously rinsed with *Purified Water*. Place the containers on the tray of the autoclave.

Place the tray in the autoclave containing a quantity of water such that the tray remains clear of the water. Close the autoclave, and carry out the following operations:

1. heat the autoclave to 100° and allow the steam to issue from the vent cock for 10 minutes;
2. close the vent cock and raise the temperature from 100° to 121° at a rate of 1° per minute;
3. maintain the temperature at 121 ± 1° for 60 ± 1 minutes;
4. lower the temperature from 121° to 100° at a rate of 0.5° per minute, venting to prevent a vacuum;
5. do not open the autoclave before it has cooled down to 95°;
6. remove the containers from the autoclave using normal precautions, place them in a water bath at 80°, and run cold tap water, taking care that the water does not contact the loose foil caps to avoid contamination of the extraction solution;
7. cooling time does not exceed 30 minutes.

The extraction solutions are analyzed by titration according to the method described