Waters Certified Sample Vials

INTRODUCTION

Glass is classified into types suitable for specific uses. Standards that govern these classifications include ASTM, EP (European Pharmacopoeia), and USP. These classifications in brief are:

USP Type I Borosilicate Glass

The least reactive glass available for containers. Surface treatment is usually not required; however surface enhancement may become important for small containers because the ratio of container surface to the volume of the container.

USP Type II De-alkalized Soda-lime Glass

Higher levels of sodium hydroxide and calcium oxide; less resistant to leaching than Type I glass. USP Type III Soda-lime Glass is acceptable. Acceptable for packaging of dry powders and liquids that are insensitive to alkali.

USP Type NP Soda-lime Glass

General purpose glass for non-parenteral applications where chemical durability and heat shock are not factors.

Chemical Composition Type I of Glass

There are different coefficient of expansion glass in Type I classification. Composition and free ion content of two of these glasses are shown in the tables below. Slight variations can occur depending on where the raw materials are sourced.

Chemical composition (main components in approx. wt %)

	SiO_2	B_2O_3	Al_2O_3	TiO_2	Fe_2O_3	Na_2O	K_2O	BaO	CaO
Amber 51-expansion	69	10	6	3	1	6	2	2	0.5
Clear 33-expansion	81	13	2	–	–	3.5	0.5	<0.1	<0.5

Free ion content

Ions	Na	K	Ca	Mg	Al	Fe	Ba	Zn	Mn	Si
Amber (51)	0.4–1.4	0.02	0.1	ND	0.5	ND	0.2	ND	ND	5
Clear (33)	0.3–0.5	ND	ND	ND	ND	ND	ND	ND	ND	ND

ND = non-detectable results in µg/mL.

Other factors pertaining to the sample must be considered before choosing the vial. Samples that are light sensitive should be stored in amber vials. The transmission curves for the clear and amber vials are shown below.

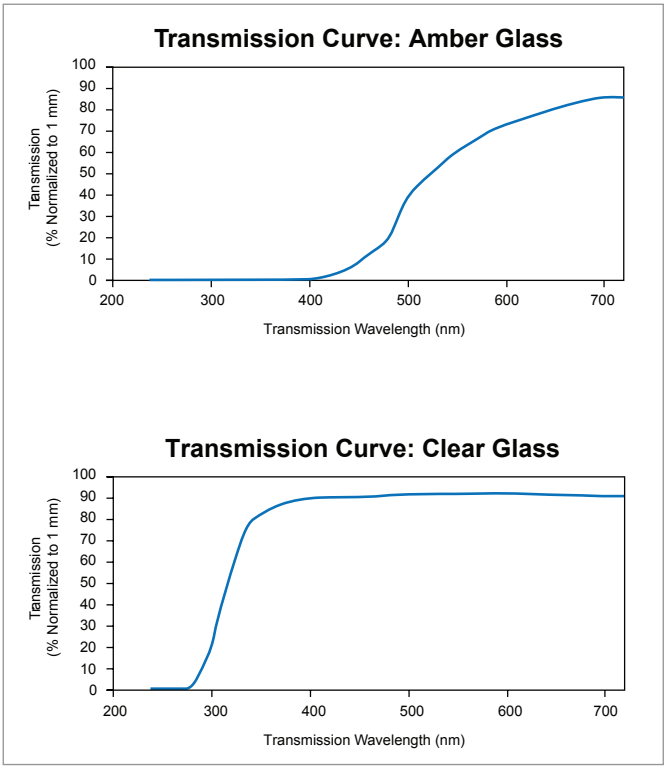

Figure 1. Transmission curves for Amber Glass and Clear Glass vials.

WATERS GLASS VIALS

Waters™ glass vials are made from USP Type I Glass. Glasses in the USP Type I classification are borosilicate glass with superior chemical resistance. This class of glass represents the least reactive glass containers available. Typically, this glass can be used for most applications, including packaging for parenteral and non-parenteral products. Type I Glass may be used to package acidic, neutral, and alkaline products. Type I Glass can be subject to chemical attack under certain conditions, so container selection must be made carefully for very low and very high pH applications.

Clear Glass Vials can be made from different coefficient of expansion glass; most notable 33-expansion and 51-expansion glass. All Waters Clear Glass vials are made from of 33-expansion glass. The alkali content is low and 33-expansion glass has very low free ion content.

Amber Vials are made from 51-expansion glass which has a different chemical composition and higher free ion content. Waters distributes the same vials (same chemical composition) around the world. Some distributors of vials source locally distributing different glass in different geographies. If they do this, their product has different chemical composition in different regions.

DEACTIVATED GLASS

Waters offers most of the glass vials line in a deactivated form. The vials are treated with a reactive organosilane in a gas phase to produce a hydrophobic surface. This treatment changes the contact angle of solutions coming into contact with the surface. Deactivated vials can be stored indefinitely.

Glass has a polar surface (Si-O-H). If the analytes in the sample can interact or bind to the glass; this may affect quantitation. Deactivated glass vials would be a better choice.

The silane can be stripped off the surface of the glass by strong solutions of aqueous base stored for an extended period. For storage of samples in strong basic solution for an extended period, plastic vials would be a better choice.

VIAL CLOSURES

Vials are available in three closure types; crimp, snap, and screw cap. The closures have advantages and disadvantages.

Crimp Caps squeeze the septum between the rim of the glass vial and the crimped aluminum cap. This forms an excellent seal preventing evaporation. The septum stays seated during piercing by the autosampler needle. The crimp cap vial requires crimping tools to carry out the sealing process. For low volume settings, manual crimper tools are the choice. For high volume settings, automated crimpers are available.

Snap Caps are an extension of a crimp cap system of sealing. A plastic cap is stretched over the rim of the vial to form a seal by squeezing the septum between the glass and the stretched plastic cap. Plastic has memory and wants to return to the original dimension. This tension in the cap to return to original size is the force forming the seal between glass, cap, and septum. The advantage to plastic snap cap is that no tools are required for assembly.

- Snap Caps are a compromise sealing system.
- If the fit of the cap is very tight, they are hard to apply and may be subject to cracking.
- If the fit is too loose, the seal is not very good, and the septa may be subject to dislodging.

Screw Cap Vials are universal. Screwing the cap applies a mechanical force that squeezes the septum between the glass rim and the cap. Screw caps form an excellent seal and mechanically hold the septum in place during piercing. No tools are required for assembly.

LectraBond Screw Caps – Waters offers a screw cap with the PTFE/Silicone septum bonded to the polypropylene cap, using a non-solvent bonding process. This bonding technology is designed to keep the septum/cap together during shipment and assembly onto vials. The bond will aid in preventing dislodging of the septum during use, but the primary sealing mechanism is the mechanical force applied by tightening the cap to the vial.

Note: For cap tightening, this is the mechanism that forms the seal and holds the septum in place during needle insertion. There is no need to overtighten the cap, this can compromise the seal and lead to dislodging. The septum begins to cup or indent when you begin to overtighten.

Cap design	Strength design	Comment
Crimp	Excellent seal	Requires tools
Snap	Moderate seal, fast	No tools, some cap cracking
Screw	Excellent seal	Universal

Table 1. Types of vial closures.

CHOOSING THE RIGHT SEPTUM

Waters offers several choices for septum. The following are the selection with recommendation for usage:

Material	Compatibility	Recommendations	Self sealing
PTFE	All solvents	Suitable for one injection	No
PTFE/Silicone	PTFE is resistant until punctured after first injection	Recommended for multiple injections and sample storage	Very good
PTFE/Silicone Pre-slit	Same	Very good for multiple injections, eliminates vacuum formation or pressure, enables accurate volume draw in autosampler, prevents coring with bottom draw needles	Good

Table 2. Septum selections and user recommendations.

WATERS CERTIFIED VIALS FOR GC AND LC APPLICATIONS

In 2004, Waters introduced a line of LC/GC Certified Vials. We understand the demands of analytical instrumentation for dimensions and chemical cleanliness.

DIMENSIONS

Waters vials are held to tight dimensional tolerances, from Type 1 pharmacopoeia grade glass. Waters understands the importance of dimensional tolerance in autosamplers. The bottom thickness and depth are closely controlled to prevent needle damage.

For dimensional testing we measure:

- Height
- Width
- Neck
- Thread
- Bottom thickness
- Roundness

The bottom thickness and rounding are very important specifications. The needle is traveling a certain distance as programmed in the autosampler. The bottom thickness must be controlled to prevent the needle from crashing into the vial bottom. The roundness specification on the vial is important to locate the septum in the center of the vial for piercing.

CHEMICAL TESTING – CLEANLINESS

Septum – The PTFE/silicone septa is tested by Headspace GC to verify the septa has been properly cured. The test is designed to look for the presence of low MW silicone polymer (DP2, DP3, DP4...), volatile silicones that could surface in chromatography.

Vials are formed into shape from glass tubing. In production of tubes, surfactants are used to prevent scratches and reduce breakage.

In the vial forming process, lubricants are used on the machinery.Waters has looked at the chemical composition of these surfactants and lubricants, and developed chromatographic tests to screen for their presence at trace levels. In the HPLC test for Certified Vials, we key in on compounds that can be detected at the ppb level. If the glass processing has not been carried out properly, residue will be detected, and the lot of vials rejected. Waters has rejected vials based on testing.

We have also looked at antistatic and antioxidant agents that may be used in the production of plastics for packaging and film (wrap). We screen the packaging materials by an HPLC test. This test can detect these agents in the packaging should they be present. All the tests, other than the headspace GC, are run on a Waters HPLC, using a Symmetry™ C_{18} Column, UV detection, and Empower™ Software.

CARE AND HANDLING

Waters has certified the dimensions and cleanliness, now it is up to you to keep the vials clean.

STORAGE CONSIDERATIONS FOR SILICONE/PTFE:

Silicone rubber can readily absorb chemicals from the environment. Care needs to be taken in storing caps prior to use.

Recommendations include:

- Don't store the septa in locations where chemical vapors are present for prolonged periods.
- Do not write on the sealed bag with a marker. Solvent vapors will penetrate the bag and be absorbed in the silicone rubber.
- Close a partially consumed bag to limit exposure for prolonged periods.

GLASS

The last step in the glass process is firing the vial at 550 °C. The vials are immediately moved into a clean room and are packaged. The packaging materials consist of very clean film, free of antistatic and slip agents. The surface of glass is very active. Partially opened packs of vials should be covered to keep them dust free and dry.

Waters

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