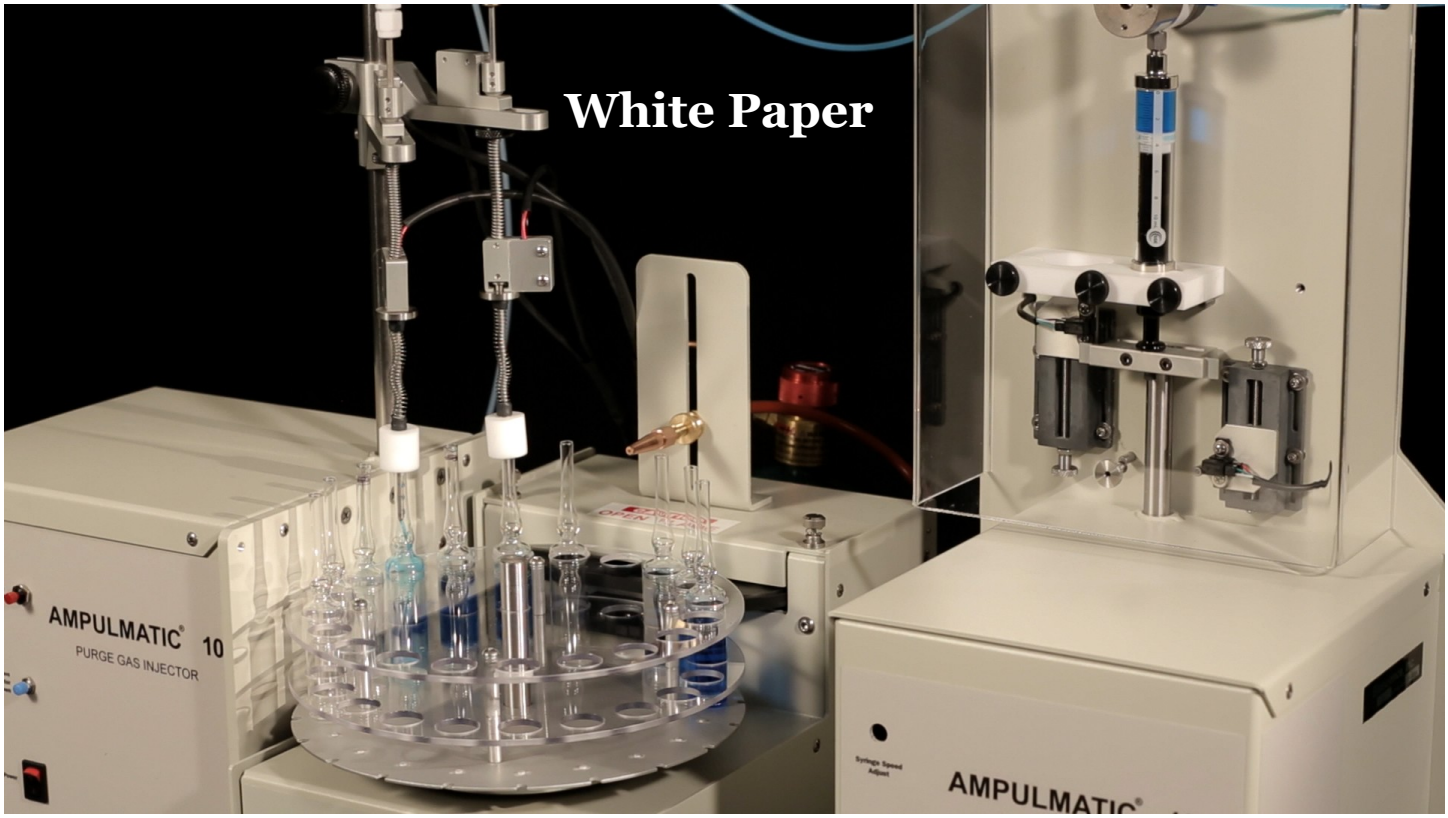

Sealing Ampules
That Contain
Unique
or
Difficult Solutions

Bioscience, Inc.
Environmental Products & Services

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Sealing Ampules That Contain Unique or Difficult Solutions

Nearly any liquid may be packaged and sealed in glass ampules, but some liquids require special procedures to ensure:

- A uniform, strong seal,
- Operator safety, and
- Minimal deterioration of equipment

This paper discusses the issues associated with sealing ampules that contain unique or difficult solutions. Bioscience, Inc. has performed ampule sealing experiments with a variety of liquids. This paper will discuss the sealing of:

- Volatile or Flammable Solvents
- Corrosive Solutions
- Chlorinated Organic Solutions

Volatile or Flammable Solutions

When sealing ampules containing volatile and flammable solutions, the following issues may be encountered:

- Burning of the solvent,
- Bubbling of the glass ampule seal,
- And blowout of the glass ampule seal.

Bubbling and blowout of the seal are caused by the increase of vapor pressure within the glass ampule while it is being sealed.

Volatility is the tendency of a substance to vaporize, and is directly related to its vapor pressure. The higher the vapor pressure of a substance at a given temperature, the higher the volatility. Therefore, the high vapor pressure of volatile substances can make sealing difficult. As the ampules are heated to create a seal, the temperature of the liquid increases, thus also increasing vapor pressure.

General Recommendations

1. When sealing volatile or flammable solvents, the use of long-necked ampules is recommended.

2. Ampules should be inspected prior to use to remove ampules with cracks or other flaws that might cause breakage and spilling of the contents.

3. Care should be taken when sealing volatile or flammable solvents. Always perform a test run with such solvents using the smallest volume of solvent possible to minimize danger to the user. The use of a safety hood or shield is strongly recommended.

Ampule Sealer Adjustment

In some cases, minimizing the sealing time (dwell time of the ampule within the flame) and/or reducing the flame temperature may eliminate bubble formation and/or blowout.

Purge Gas Injection

Alcohols, crude oils, and alkanes such as hexane can be sealed at ambient temperatures with purge gas injection. Injecting an inert purge gas (i.e. argon or nitrogen) into the ampule after filling displaces both oxygen and solvent vapor from the ampule, reducing vapor pressure and flammability.

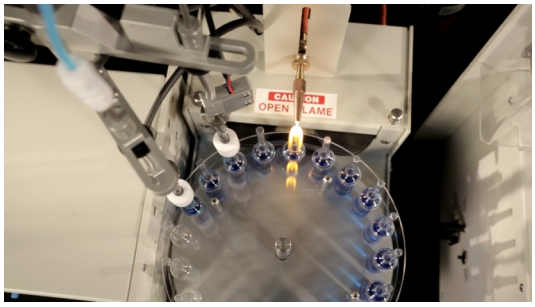
Chilling of Solvents

Very volatile and flammable solvents may need to be chilled to reduce flammability and/or vapor pressure sufficiently to ensure safe sealing. The solvents may be chilled by immersion in cold baths of various types prior to flame sealing (see Table 1).

Table 1: Solvent Chilling

Bath components	Concentration of *	Temperature °C
Ice/water/NaCl*	Excess	-10 to -15
Ice/water/$CaCl_2$*	Excess	-29
Dry ice*/ethanol or isopropanol	Excess	-72 to -78
Dry ice*/ethylene glycol	Excess	-15

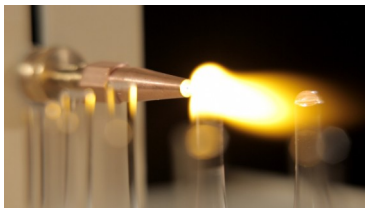
If a solution is stable such that components will not separate on chilling, the chilled solvent can be dispensed into ampules. Unstable solutions may need to be dispensed into ampules first, then chilled prior to placing in the carousel. **Note:** the syringe used in our liquid filler will not seal properly if the liquids are chilled below ~5°C. A chilling coil immersed in a cooling bath may be usable with the liquid filler (inserted in the flow path between the syringe and the ampule filler head). With or without purge gas injection, the filled ampules should be advanced into the flame sealing position as rapidly as possible to minimize warming of the solvent. Keeping the solvent temperature consistent is important to obtain consistent seals.



When setting up your ampule sealing equipment, make initial adjustments with empty ampules or ampules containing non-flammable solvent. Final adjustments are then made with chilled solvent to ensure good sealing.

Very cold solvents will condense water or even form ice on the outside of the ampule. This condensation may interfere with the uniform spinning of the ampules and result in poor seal quality. Use of an adapter (for example, a PTFE rod drilled to hold 1-5 mL ampules in a 10 mL carousel) insulates the ampule and prevents condensation from affecting ampule spinning.

Corrosive Solutions:



Corrosive solvents can damage mechanical components. Communication with Bioscience prior to purchase regarding specific solvents to be used allows us to replace parts which are less resistant in the standard equipment with more resistant materials. A higher grade of stainless steel or titanium may be required for certain components. Note that chlorinated organic solvents degrade in the flame and may create a corrosive atmosphere due to hydrochloric acid formation.

High in Salt Solutions:

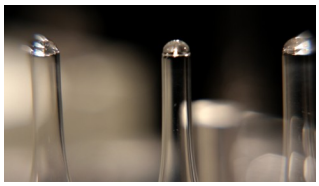
High salt concentrations may clog the dispensing syringe or tubing of the Liquid Filler accessory. The solution should not precipitate and regular flushing may be required to maintain flow.

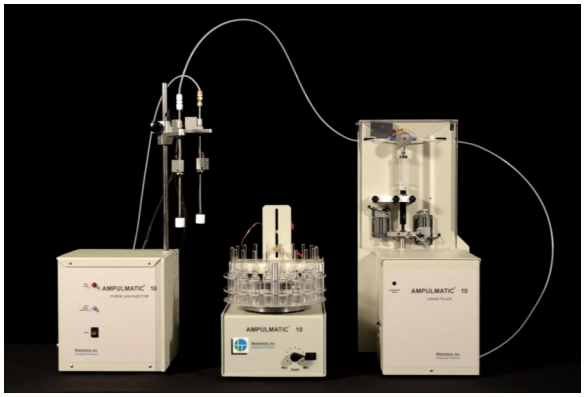
Higher Viscosity Solutions:

Higher viscosity solutions can be dispensed using the Ampulmatic-10 liquid filler. Viscosity primarily affects the rate of flow through the inlet tubing and thus the rate of fill of the syringe. Larger diameter tubing can be used to increase the flow rate. The syringe must be able to fill completely in less than the time required for sealing the ampule to maintain volumetric accuracy.

Materials That Require Oxygen Exclusion:

Materials that degrade due to oxidation will benefit from the purge gas injection of nitrogen or preferably argon. Although the purging does not eliminate oxygen, oxygen concentrations in the headspace of the ampule can be reduced to less than 1%. Consult Bioscience for ways to minimize residual oxygen.





Conclusion

Bioscience, Inc. personnel have worked with customers to solve a variety of difficult applications. If we do not have experience with a particular application, we can perform testing in our laboratory with customer submitted ampules and/or filling solutions. We also can provide equipment for customers to test and develop their own protocols.

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