SOP: Degassing a Solution by Helium Sparge

Approvals

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1. **Purpose**
   1.1. Remove dissolved atmospheric gases from a solution by means of sparging with helium.
2. **Scope and Applicability**
   2.1. Applicable to preparing solvent solutions for use in High Performance Liquid Chromatography (HPLC). Solution volume is limited to the range of 25 mL to 800 mL.
3. **Summary of Method**
   3.1. Helium gas is delivered from a compressed gas tank to a porous metal sparger that is placed in a bottle of solution. Helium is bubbled through the solution for 15 minutes.
4. **References**
   4.1. Airgas® Operation and Safety Instructions For Specialty Gas Regulation Equipment, form #320-517 Rev. 5/03
5. **Definitions**

Helium sparge   Using a stream of helium bubbles to sweep dissolved air out of liquids (helium is virtually insoluble in most HPLC solvent solutions, so very little helium replaces the air)

HPLC   High Performance Liquid Chromatography

PSI   Pounds per Square Inch

6. **Precautions**
   6.1. A helium tank is compressed to pressures up to 2000 PSI. A sudden release of pressure can cause serious damage to personnel and equipment. Handle the helium tank and gas regulator with care and wear eye protection.
   6.2. Helium sparging entails some risk of changing the composition of an HPLC solvent solution by selectively evaporating the more volatile components. Avoid an excessively vigorous flow of helium.
7. **Responsibilities**
   7.1. It is the responsibility of the course instructor/lab assistant to ensure that this SOP is performed as described and to update the procedure when necessary.
   7.2. It is the responsibility of the students/technician to follow the SOP as described and to inform the instructor about any deviations or problems that may occur while performing the procedure.

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Equipment and Materials

    7.3. Laboratory grade (99.9+%) helium in a compressed gas cylinder (helium tank)

    7.4. Airgas® gas cylinder regulator pre-connected to the helium tank

    7.5. Porous metal sparger

    7.6. Flexible gas tubing

    7.7. Laboratory bottle containing HPLC storage solution (e.g. 50% MeOH/H_2O or 50% Acetonitrile/H_2O).

    7.8. Solution to be sparged (25 to 800 mL)

    7.9. Empty laboratory bottle (50, 100, 250, 500, or 1000 mL)

    7.10.   Two-hole stopper that is sized to the empty laboratory bottle (size # 1 for 50 or 100 mL bottle, size #6.5 for 250, 500, or 1000 mL bottle)

    7.11.   Laboratory grade water in a wash bottle

    7.12.   Waste beaker

    7.13.   Timer

8. Procedure

    8.1. Verify that all personnel in the area are wearing eye protection.

    8.2. Connect the sparger to the helium tank if it is not connected already:
      (See Figure 1 for location of valves.)

        8.2.1. Turn the gas pressure off by turning the regulator valve <u>counter-clockwise</u> for two full turns (in the direction marked "DECREASE").

        8.2.2. Close the delivery valve by turning it clockwise until it stops.

        8.2.3. Close the tank valve by turning it clockwise until it stops.

        8.2.4. Connect one end of the flexible gas tubing to the gas regulator.

        8.2.5. Connect the other end of the flexible gas tubing to the porous metal sparger.

        8.2.6. Insert the gas tubing nearest the sparger through one of the stopper holes.

        8.2.7. Place the sparger in the HPLC storage solution bottle and cap with the stopper.

    8.3. Open the helium tank with the flow of helium turned off:

        8.3.1. Verify that the gas pressure is turned off by turning the regulator valve <u>counter-clockwise</u> for one half turn (in the direction marked "DECREASE"). The valve knob should turn freely.

        8.3.2. Close the delivery valve by turning it clockwise until it stops.

        8.3.3. Slowly open the tank valve by turning it counter-clockwise.

    8.4. Dispense the solution to a correctly sized laboratory bottle:

        8.4.1. Select an empty laboratory bottle that has 20%-50% more volume than the quantity of solution to be sparged. (Note: additional volume is needed as head space to accommodate the sparger and bubbles without spilling.)

        8.4.2. Measure the desired amount of solution and transfer it to the selected bottle. Verify that the bottle is at least 50% full and no more than 80% full.

    8.5. Place the sparger in the solution bottle:

        8.5.1. Remove the sparger from where it was stored in the HPLC storage solution bottle.

        8.5.2. Rinse the sparger with water from a wash bottle over a waste beaker.

        8.5.3. Place the sparger into the solution bottle and cap with the stopper.

        8.5.4. Verify that the sparger is submerged in the solution and that the unused stopper hole is unobstructed.

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8.6. Turn on the flow of helium and set the deliver pressure to 3 PSI:

    8.6.1. Verify that the gas pressure is turned off by turning the regulator valve <u>counter-clockwise</u> for one half turn (in the direction marked "DECREASE"). The valve knob should turn freely.

    8.6.2. Slowly open the delivery valve by turning it counter-clockwise.

    8.6.3. Watching the delivery pressure gauge, slowly increase the gas pressure by turning the regulator valve <u>clockwise</u> (in the direction marked "INCREASE") until the delivery gauge reads 3 PSI.

    8.6.4. Observe bubbles rising from the sparger. Verify that there is sufficient head space in that bottle so that solution is not expelled from the bottle.

8.7. Sparge the solution for 15 minutes.

8.8. Turn off the flow of helium:

    8.8.1. Turn the gas pressure off by turning the regulator valve <u>counter-clockwise</u> for two full turns (in the direction marked "DECREASE").

    8.8.2. Close the delivery valve by turning it clockwise until it stops.

8.9. Store the sparger:

    8.9.1. Remove the sparger from the solution bottle and cap the solution bottle.

    8.9.2. Rinse the sparger with water from a wash bottle over a waste beaker.

    8.9.3. Place the sparger in the HPLC storage solution bottle and cap with the stopper.

8.10. Close the tank valve by turning it clockwise until it stops.

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Document Number: QCB 6
Revision Number: 0
Effective Date: 25SEP13
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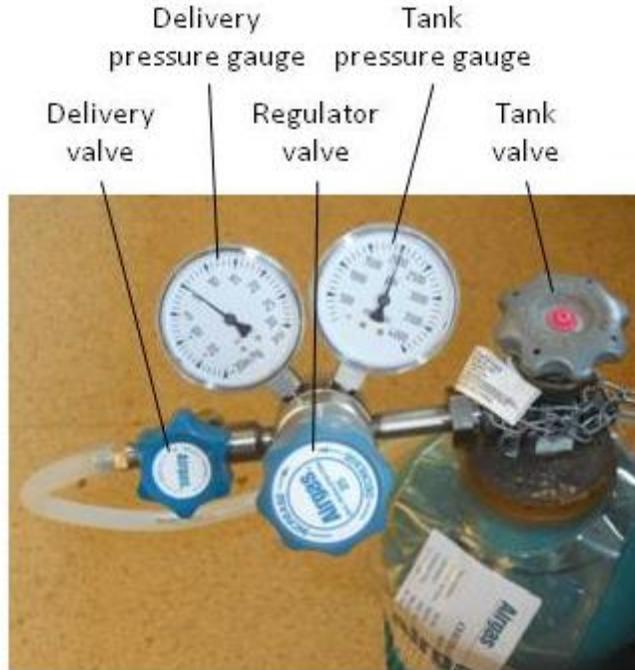
9. Attachments

Figure 1. Helium Tank and Gas Regulator

Figure 2. Porous Metal Sparger

10. History

Revision Number	Effective Date	Preparer	Description of Change
0	09/25/2013	John Buford	Initial release