Maintenance Tips for Users of the MEINHARD® Concentric Glass Nebulizer

(A PDF version of these maintenance tips is available here.)
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Your MEINHARD® nebulizer reflects the exacting standards in design and manufacturing that have made it the choice of the industry worldwide. Properly maintained, this nebulizer should yield stable performance indefinitely. However, failure to apply a few simple preventive measures can lead to obstructed gas and liquid passages in the nebulizer, which can seriously impair operation. We recommend that you follow the maintenance tips outlined here for long-lasting and trouble-free performance. We also include a diagnosis and treatment scheme [1] to help restore inoperative (but undamaged) nebulizers (see Figure 2 at end of booklet.). Refer to Figure 1 below for nebulizer configuration. We provide this information as a courtesy to our customers.

WARNING!
Meinhard Glass Products assumes no responsibility for accidental damage resulting from application of the recovery procedures.

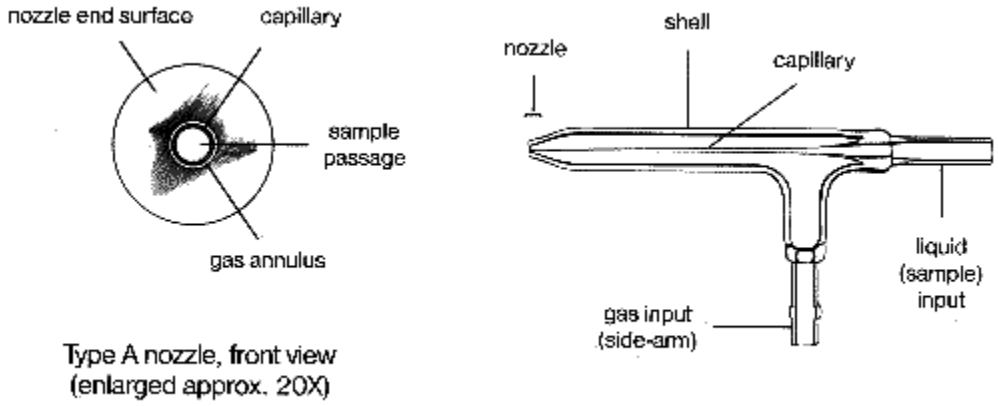

Figure 1. MEINHARD® Nebulizer Configuration

Preventative Measures

Blockage in the nebulizer is caused generally by either particulate matter (from the sample or the carrier gas) or chemical deposits. It normally occurs in the nozzle where the flow passages are extremely small. Constriction is greatest in the annular gas channel between the tip of the capillary and the taper of the nozzle. In some nebulizer models, this annular gap can be as small as 10-15 µm. See Figure 1.

Tip #1:

Filter the carrier gas. Install low-impedance in-line gas filters to prevent particles from being carried into the nebulizer and lodging in the gas annulus. This is especially important when polytetrafluoroethylene (PTFE-Teflon®) tape has been used in the gas line plumbing. Shreds of this tape have been found wedged in the gas annulus and have resulted in drastic reductions in performance [2]. For the same reason, avoid using PTFE or other friable sealants at the gas connection to the nebulizer.

Tip #2:

Filter the sample. The sample capillary is more tolerant of particulate matter than the gas annulus. In high sample uptake models especially, the capillary will carry visibly turbid suspensions. We suggest, however, that you filter or centrifuge the sample if the solids are not of analytical importance. Particulates and colloids of a polar nature such as silica, peptides, polyvalent metal hydroxides, and others tend to build up on the (polar) glass and impede the fluid flow. In some instances, you can prevent deposition by adjusting the pH of the suspension away from its (presumed) isoelectric point.

Tip #3:

Rinse your nebulizer. It is especially important to rinse the nebulizer before turning the gas off. Depending on the chemistry of your samples, it may also be advisable to rinse periodically throughout the testing. Solids may deposit in the nozzle as sample solvent evaporates, further constricting the flow passages and reducing the signal. Rinsing will minimize or eliminate these deposits. Gas flow through most nebulizer models creates venturi suction at the capillary tip that can be used to draw rinse liquid through the capillary. Follow the testing of any salt solution promptly with a chemically compatible rinse consisting only of volatiles. (This is not as necessary in flow injection analysis systems.) A low-pH (acidic) sample should be followed by a low-pH rinse; a high-pH sample by a high-pH rinse; an organic sample by an appropriate solvent. Rinse finally with pure water and/or isopropyl alcohol. Allow the nebulizer to dry before turning off the gas. Also, make sure that the liquid feed is disconnected or arranged to prevent siphoning into the nebulizer while the gas is off.

Tip #4:

Do NOT use ultrasonic cleaning. Sympathetic vibrations may be set up in the capillary causing it to bounce against the inside of the nozzle and chip. Nebulizer performance can decline severely as a result.

Recovery Methods

If you find that solids inside the nebulizer are interfering with performance, you generally can remove them by means of the steps outlined below. Full function usually can be recovered. This sequence is diagrammed in Figure 2.

1. Preliminary Examination:

Disengage the nebulizer from its mounting and examine it under 20X or 30X magnification. If a particle appears to be wedged in one of the channels, go to Step 2. If solids are present in the capillary but there is still a passage through them, go to Step 3. If solids have completely blocked the capillary, go to Step 6. If the nozzle is encrusted with crystalline deposits, go to Step 4. If no foreign matter can be seen, go to Step 5. In all cases, discontinue treatment when you have removed the obstruction.

NOTE: Rinse the nebulizer after any treatment that introduces caustic or corrosive cleaning agents into the shell and/or capillary. Make sure that the final rinse is with isopropyl alcohol to speed the drying process. Introduce a rinsing agent into the shell, either from the gas input or the nozzle (a squeeze bottle works well in both cases). Fill all areas previously exposed to corrosive solutions. Attach pressurized gas to the side-arm to expel the liquid. Inject more rinse solution into the liquid input while the gas is flowing and allow venturi suction to draw it through the capillary. Repeat the treatment if you think it necessary. Finally, dry the nebulizer completely.

NOTE: If you have an A-type nebulizer, avoid touching the capillary tip when injecting solutions into the shell from the nozzle. The capillary is flush with the nozzle surface.

2. Particles:

These operations are ranked in order of increasing aggressiveness. We recommend that you start with the gentlest. Some of these methods may take a little practice before they feel comfortable.

a. Tap the liquid input of the nebulizer gently against a wood surface (or surface of comparable hardness) to jar the particle loose. This helps the particle to move in the direction of increasing

inner diameter. Repeat the tapping as necessary to work the particle toward the appropriate exit orifice. Avoid extremely harsh tapping.

b. Apply compressed gas (15-30 psig) to the nozzle, forcing the gas backwards through the annulus and the capillary (back-flushing). Make sure you hold the nebulizer securely during this operation. Tap or flick the shell soundly with your fingernail a few times. If this fails to dislodge the particle, close off the liquid and gas input tubes with your fingertips. When the pressure builds up, "pop" a fingertip quickly off the appropriate orifice (if something is wedged in the gas annulus, "pop" your finger off the gas input; if in the capillary, off the liquid input). The sudden expansion of gas should help jar the particle loose in the direction of increasing inner diameter. Try to orient the nebulizer so that gravity assists you.

c. Force isopropyl alcohol backwards through the nozzle in an attempt to float the particle out through the larger gas and liquid input tubes. Use a squeeze bottle or plastic dropper with a tip that will form a good seal over the nebulizer nozzle. Then blow the alcohol through the input tubes using compressed gas, or blot onto lint less tissue (e.g., "Kimwipe").
A variation of this procedure works best if (1) you know which passage the particle is in and (2) your nebulizer is a type C or K with a recessed capillary. Inject 1/4" to 1/2" of solvent into the shell through the nozzle or the gas input tube. Close off the nozzle with a fingertip. (**Caution:** Use protection if the solvent is caustic.) Apply pressurized gas to the passage that does not contain the particle. Pressurized solvent will force its way out the other channel in the direction of increasing diameter, hopefully carrying the particle along with it.

d. If the particle remains and you believe that it might be a shred of PTFE tape from the gas line, immerse the nebulizer nozzle in hot water and apply gentle gas pressure to the side-arm. The hot water "relaxes" the polymer and allows it to be forced out of the nozzle.

e. To loosen especially stubborn particles such as silica, use a solution of 3%-5% hydrofluoric acid (HF). (**Caution:** Use appropriate protection and care when handling HF.) HF breaks the bonds between glass and other substances. Introduce the acid into the nozzle from a plastic dropper. Drain after 5-10 seconds and rinse with pure water and/or isopropyl alcohol. Examine the nebulizer under magnification. This process can be repeated several times without significantly altering the performance of your nebulizer. However, longer soaks (e.g., 5 minutes) and higher HF concentrations will reduce the operating gas pressure. Dry the nebulizer thoroughly after the final treatment.

f. If the particle still remains and is believed to be organic (e.g., a cellulose fiber), go to Step 5.

g. If the particle is located in the capillary and still does not move, go to Step 7.

3. Solid Deposit in Sample Capillary:

This step assumes that a passage still exists through the contaminating material. If the capillary is plugged completely, go to Step 6.

a. Try to deduce from prior use the chemical nature of the deposit. Select the solvent most likely to dissolve it. Inject the solvent into the nozzle with a plastic dropper or squeeze bottle until the affected area is filled. Expel the solvent with compressed gas. Refill and expel the solvent repeatedly. Examine the nebulizer under magnification. If the material is gone, rinse the nebulizer with isopropyl alcohol and dry thoroughly.

b. If the residue remains, fill the affected area with solvent and warm the nebulizer under a heat lamp. Place it 20-25 cm (8-1 0 in.) away from the heat. Heat as long as necessary. The solvent may boil out of the capillary loosening the residue and perhaps carrying some of it away. This does not harm the capillary. Refill and repeat the process as necessary. You may substitute microwave heating for radiant heating. A treatment of up to 15 seconds may be sufficient but this should be determined empirically for your equipment. Cover the nebulizer(s) with paper towels to trap any particles forced out by the solvent vaporization. (**Note:** The microwave may be used to dry the nebulizer any time this is required.)

c. To loosen especially stubborn (hydrophilic) deposits such as silica or barely soluble metal salts, use a solution of 3%-5% hydrofluoric acid (HF) (as in Step 2e above). (**Caution:** Use appropriate protection and care when handling HF) The HF breaks the bonds between glass and other material, even in the case of baked on gold and platinum. Introduce the acid into the nozzle from

a plastic dropper. Expel after 5-10 seconds and follow with a rinse of pure water and/or isopropyl alcohol. Examine the nebulizer under magnification. You can repeat this process several times without significantly altering the performance of the nebulizer. However, longer soaks (e.g., 5 minutes) and higher HF concentrations will reduce the operating gas pressure. Dry the nebulizer thoroughly after the final treatment.

d. If the deposit remains and you believe it is organic, go to Step 5.

4. Crystalline Encrusted Nozzle:

Accumulated salt deposits occur most frequently with high-concentration samples or when the tubing arrangement permits siphoning of the liquid when the gas is not flowing. Salt deposition is controlled with appropriate rinsing.

a. Immerse the nozzle in a rinse solution. Warm the solution for stubborn deposits. Follow with a rinse of pure solvent, then isopropyl alcohol and dry thoroughly.

b. If the deposit remains after prolonged soaking, apply pressurized gas at the appropriate input(s) to help force the deposit away. Return to Step 4a above.

c. Apply the techniques outlined in Step 3c (HF) if the above treatments fail to remove the entire residue.

5. Organic Matter:

Immerse the nozzle of your nebulizer in a hot, concentrated nitric acid. Hold the temperature at about 100ºC. (**Caution:** This solution is corrosive. Please use it with appropriate safeguards.) Allow the solution to rise into the passages of the nebulizer until the affected area is filled. Expel and replace the solution at intervals until the deposit is gone. Rinse the nebulizer thoroughly with water, then with isopropyl alcohol, and dry completely.

Caution: Cleaning with chromic acid is not recommended. It may be impossible to remove all traces of chromium from the glass.

6. Plugged Capillary (fusible solids such as waxes):

Follow these steps when no passage remains through the deposit:
Carefully heat the nebulizer in the region of the capillary obstruction. Simultaneously, (or intermittently) apply gentle gas pressure at the sample input tube. (**Caution:** avoid overheating residues that may produce insoluble pyrolysis products.) Stop treatment when you have opened a passage through the blockage. Return to Step 3 (Capillary Deposit) or Step 5 (Organic Matter) to complete the cleaning.

7. Firmly Lodged Particle in the Capillary:

WARNING!
This procedure places the nebulizer at substantial risk!
Resort to it only when all other methods have failed!

Insert a fine (7-8 mil) piano wire into the capillary from the nozzle end of the nebulizer. Gently push against the particle, in the direction of increasing diameter, until it is dislodged. Avoid pushing hard enough to buckle the wire. This can break the capillary. Such damage will be permanent and could have

a drastic negative effect on the nebulizer's performance. Remove the wire and back flush with compressed gas. Do not attempt to insert the wire (or any other object) into the gas annulus.

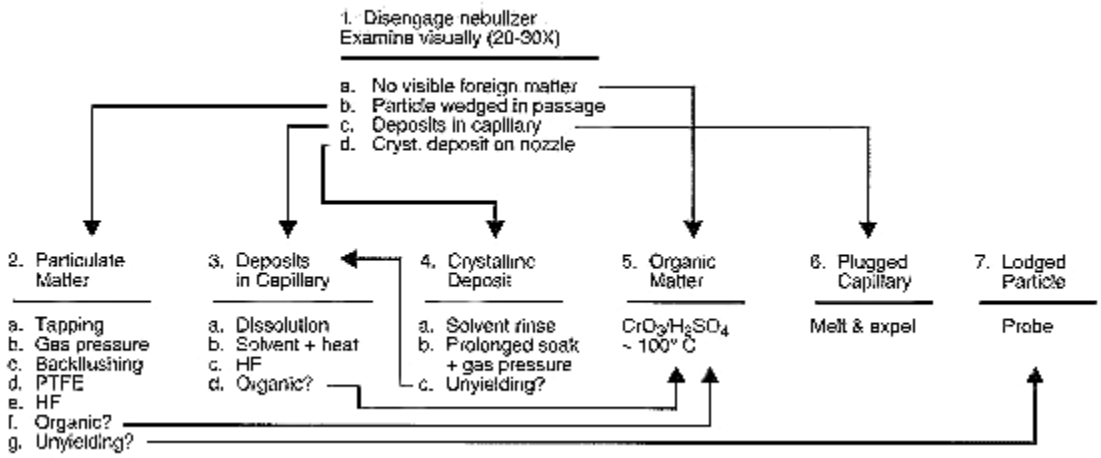

Figure 2. Cleaning Strategy for the MEINHARD® Nebulizer

[1] Updated from a paper originally presented at the 1980 Winter Conference on Plasma Spectrochemistry, San Juan, PR., and reproduced in the ICP Information Newsletter, Vol. 12, No. 9, 677, (1987). R. M. Barnes, Ed.

[2] Private communication. A number of comments and suggestions related to concentric nebulizer maintenance have been received over the past several years. To cite these sources individually is not possible, but their contributions are gratefully acknowledged.