

## Cone Care and Maintenance

### Introduction

All sample and skimmer cones, regardless of ICP-MS manufacturer or design serve the same function. They are the differential apertures separating the sample at atmosphere from the mass filter and detector(s) at high vacuum. Though the fundamental dimensions are all essentially the same (sample cone with shallow angle, skimmer cone with acute angle), each manufacturer has designed the cone's critical dimensions to their own unique specifications (orifice diameter, hole depth, material composition, etc.). The actual cone design may vary significantly from one instrument manufacturer to another. The analytical zone, the area most in need of maintenance displays the most significant differences. These differences, together with sample type (ground water, soil digests, sea water, etc) contribute to the rate at which oxides and salts accumulate in and around the cone orifice. They also affect the rate of cone degradation and influence the cleaning methodology.

Ultimately, the quality of your data is the best diagnostic tool for determining when to clean or change your cones. However, if you notice significant buildup on the cone(s), which appear to be occluding the cone orifice, cone removal and cleaning is warranted. All instrument manufacturers prescribe a daily routine to evaluate the status of the system prior to an analytical run. These fast and easy tests can also be used to qualitatively evaluate cone condition.

#### **Clogging/Blocking:**

As oxides and salts build up around and within the cone orifice they will change the gas flow dynamics and perhaps electrical characteristics of the cone interface. Symptoms may include reduced sensitivity and a degradation of signal stability. In severe cases, there may be a change in signal response across the mass range due to pressure changes (higher vacuum) between the sample and skimmer cone.

#### **Contamination:**

Like other components of the sample introduction system, the sample cones are prone to certain memory effects (eg. Li, B, Mg). This is particularly true when attempting trace analysis of elements that were previously in solution at high concentration. These effects may inhibit quality analysis well before visual buildup occurs around the analytical zone of the cones. In many if not most cases, memory effects are due to the sample introduction system (tubing, spray chamber, torch), hence thorough evaluation of all potential sources of memory effects should be done.

## Introduction (cont.)

### **Cone Degradation:**

Whether from extensive use, improper use, aggressive matrices or blunt-force trauma, over time cones will degrade beyond acceptable limits. Careful handling and proper cleaning will typically enable you to get about 500 hours (several months) of operational use from each set of nickel cones and about 1500 hours (18 months) from platinum cones. Some common symptoms of cone degradation are elevated oxide levels (eg. CeO), doubly-charged species and in severe cases poor vacuum levels.

### **Platinum vs Nickel:**

Pt-tipped cones offer greater lifetime than nickel cones (> 1500 hours) and usually require less day-to-day maintenance. Aggressive matrices (high in oxidizing conditions) may require Pt-tipped cones. Unlike nickel cones, Pt-tipped cones can be refurbished. Spectron's free refurbishing process can more than double the normal lifetime of platinum cones.

Platinum usually stays cleaner because it runs hotter and has a tighter grain. The tip can usually be reshaped several times. After extensive use, Pt (as well as nickel) will suffer from high temperature embrittlement characterized by large grain crystals that will crack when the tip is being reformed [Appendix C]. We strongly advise users not to try to mechanically alter the tip in any way because the tolerances are tight and the shape is critical to good performance. This can lead to the unnecessary, early retirement of a \$1500+ cone.

## Cleaning your cones

Whenever possible we recommend cleaning your cones under mild, non-mechanically conditions. This is especially true for platinum cones, where accidental damage is very costly. We understand however, that for some applications a refractory build up is inevitable and will require mechanical cleaning. Following are three cleaning methods. Choose the one that best suits your needs. As an alternative to the more aggressive mechanical cleaning of your platinum cones, Spectron offers a free Pt cone refurbishing service.\* Our skilled, experienced machinists can remove the most severe refractory build-up and reshape damaged Pt cones to their original specifications.

For more information please refer to: *Platinum Cone Refurbishing* in the **Library and Resources** section of our website

### **Light Cleaning – Detergent Method**

For some of you, cleaning cones is primarily a process of reducing elemental memory and improving detection limits. There is no severe build-up and what little build-up exists is easily removed.

#### **Ni – Pt/Ni Cone:**

- Soak in 1:20 Citranox:H<sub>2</sub>O for 2 hours (sonicate for last 15 min.)
- Rinse thoroughly with tap water
- Soak in dH<sub>2</sub>O for 10 min. (sonicate)
- Replace dH<sub>2</sub>O and repeat 10 min. soak (sonicate)
- Dry thoroughly (Argon or Nitrogen, if available)

If using a bath sonicator (recommended) place cones in Citranox™ solution and sonicate for the final 15 minutes of the 2 hour soaking period.\* Rinse thoroughly in tap water, then place in dH<sub>2</sub>O and sonicate for 10 minutes. Replace dH<sub>2</sub>O and repeat. Dry with clean, dust free cloth and then let air dry, blow dry with Argon or Nitrogen, or place in glassware oven (40-50°C). Always take care not to place your cone tips in harms way. For most sample cones this is not a significant problem as the base is large relative to the height. Skimmer cones however are more prone to tipping over and deforming, since they tend to have thinner walls and sharper edges.

#### **Ni/Cu – Pt/Cu Cone:**

Cleaning cones with a copper base (eg. Agilent) requires a slightly modified technique so as not to unduly wear the copper part of the cone, especially where it meets the nickel or platinum tip. Over time Citranox will attack the copper and may degrade the weld between the two metals.

- Invert cone into fixture containing filled with Citranox [Appendix B]
- Soak in 1:20 Citranox:H<sub>2</sub>O for 2 hours (sonicate for last 15 min.)
- Rinse thoroughly with tap water.
- Rinse thoroughly with dH<sub>2</sub>O (sonicate for 15 min.)
- Replace dH<sub>2</sub>O and repeat rinse (sonicate for 15 min.)

\* Soaking and cleaning times may be modified depending on need.

*The cone fixture, easily assembled from a spare cone container, enables more aggressive cleaning of the analytical zone without degrading the bimetallic weld.*

*For more information contact Spectron, Inc. 805.642.0400/800.238.8940*

### **Standard Cleaning – Acid/Detergent Method**

Mild accumulation of salts and oxides may build-up around the analytical zone of your cones. Over time this will affect the quality of your analyses. In this case we recommend a slightly more aggressive cleaning protocol.

#### **Ni – Pt/Ni Cone:**

- Soak in 1:20 Citranox:H<sub>2</sub>O for 2 hours (sonicate for last 15 min.)
- Rinse thoroughly with tap water
- Invert cone into fixture containing 2% HNO<sub>3</sub> [Appendix B]
- Let sit for 10 minutes, or place in bath sonicator
- Wipe off residue with cotton swab, then rinse in tap water
- Return to cone fixture for 10 minutes
- Rinse thoroughly with tap water
- Rinse thoroughly with dH<sub>2</sub>O (sonicate for 15 min.)
- Replace dH<sub>2</sub>O and repeat soaking/sonication
- Dry with clean, dust free cloth and then let air dry, blow dry with Argon or Nitrogen, or place in glassware oven (40-50°C).

#### **Ni/Cu – Pt/Cu Cone:**

Cleaning cones with a copper base requires a slightly modified technique so as not to unduly wear the copper part of the cone, especially where it meets the nickel or platinum tip. Over time Citranox and nitric acid will attack the copper and may degrade the weld between the two metals.

- Invert cone into fixture containing filled with Citranox [Appendix B]
- Soak in 1:20 Citranox:H<sub>2</sub>O for 2 hours (sonicate for last 15 min.)
- Rinse thoroughly with tap water
- Invert cone into fixture containing 2% HNO<sub>3</sub>
- Let sit for 10 minutes, or place in bath sonicator
- Wipe off residue with cotton swab, then rinse in tap water
- Return to cone fixture for 10 minutes
- Rinse thoroughly with tap water
- Rinse thoroughly with dH<sub>2</sub>O (sonicate for 15 min.)
- Replace dH<sub>2</sub>O and repeat soaking/sonication
- Dry with clean, dust free cloth and then let air dry, blow dry with Argon or Nitrogen, or place in glassware oven (40-50°C).

*Some of you may want to soak your **platinum** cones in a 5% or 10% HNO<sub>3</sub> solution. This should be done with great care to insure the nickel or copper base is not in contact with acid and degraded. The fixture [Appendix A] will enable you to insert the cone into solution without degrading the metal surrounding the Pt tip.*

## **Aggressive Cleaning – Mechanical Method**

### **All Cones (Ni – Pt/Ni & Ni/Cu – Pt/Cu Cone):**

- Soak in 1:20 Citranox:H<sub>2</sub>O for 1 hour (sonicate for last 15 min.)\*
- Rinse thoroughly with tap water
- Mix alumina powder with water to make a loose paste [Appendix B]
- Using felt pad apply paste to top side of cone and clean with care
- It is easier to clean the back side with a cotton swab (see image)
- Rinse thoroughly with tap water
- Soak in dH<sub>2</sub>O (sonicate for 15 min.)
- Replace dH<sub>2</sub>O and repeat soaking/sonication
- Dry with clean, dust free cloth and then let air dry, blow dry with Argon or Nitrogen, or place in glassware oven (40-50°C).

***Rinsing thoroughly in a bath sonicator is advisable. This will insure the removal of all residue accumulated during the mechanical cleaning process.***

*You may choose to skip the Citranox step, however, it will make the cleaning process easier. Cone fixture is optional*

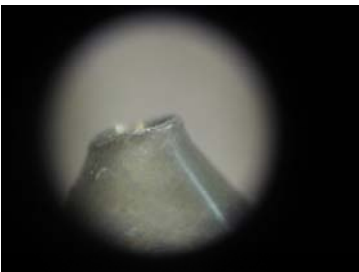
*Diamond paste and special cleaning sticks are available from Spectron for enhanced polishing of the sample and skimmer orifice.*

## Appendix A

### A Few Examples



**Typical skimmer cone with oxide buildup:** We recommend soaking this cone in Citranox™ for a few hours, then sonicating for 15 min. If this does not remove all residue follow *Aggressive Cleaning* protocol. If Citranox and sonication sufficiently clean the cone, rinse thoroughly in tap water then dH<sub>2</sub>O. Dry according to instructions.



**Delicate skimmer cone damaged in an attempt to clear orifice with metal wire:** This cone is now functionally unusable. Many skimmer cones have very thin walls with sharp edges. Any attempt to force a probe (metal, wood or plastic) through this delicate orifice may alter the cone dimensions outside manufacturer specifications



**Skimmer cone with a simple salt build up:** Sonicating in dH<sub>2</sub>O or dilute HNO<sub>3</sub> (<1%), after soaking in Citranox can usually clean these cones effectively. Dry according to instructions.

## Appendix B

### Cleaning Your Cones

#### Acid/Detergent Cleaning Method



Using this simple cone fixture it is possible to efficiently clean the cone's analytical zone without degrading the rest of the cone unnecessarily. This is particularly useful for Pt tipped cones and cones with exposed copper bases.



The cone's tip is inverted into fixture. The container can be filled with Citranox, nitric acid or a cleaning solution of your choice. Using this method, HNO<sub>3</sub> concentrations as high as 10% can be used to clean platinum cone tips without damaging the nickel or copper base. When cleaning nickel cones limit your nitric concentration to 2%.

*Some of you may choose to soak/sonicate your nickel or platinum/nickel cones directly in a Citranox solution. Though this is perfectly ok we suggest **not** placing your copper based cones (Pt/Cu, Ni/Cu) directly into Citranox or acid as this will shorten the cone's lifetime.*

## Mechanical Cleaning Method – Sample Cone

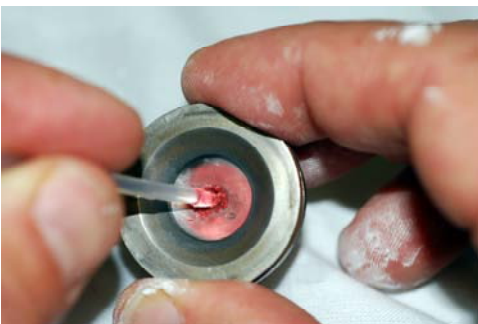
Spectron recommends pre-soaking your cones in Citranox before manually cleaning your cones (see *Aggressive Cleaning*, page 5)



After creating a light paste with fine alumina powder, polish your cone tip using a felt pad or synthetic, lint free cloth. Though the felt pad is gentle, do not apply excessive pressure directly on the tip of the cone. You want to remove build up without changing the geometry of the cone tip.



A cotton swab is very effective at cleaning the inside (concave side) of the cone. Using the same alumina paste, clean as close to the orifice as possible. By creating a point at the tip of the cotton swab it will be possible to clean residue from the inside edge of the orifice.



For optimal cleaning of the cone orifice Spectron recommends using diamond paste and a foam tipped cleaning stick. These sticks simplify the effective cleaning of the most important region of the analytical zone.



At left are the tools we recommend for mechanically cleaning your cones. For additional information send us your questions to: [Spectron@SpectronUS.com](mailto:Spectron@SpectronUS.com)



## Mechanical Cleaning Method – Skimmer Cone



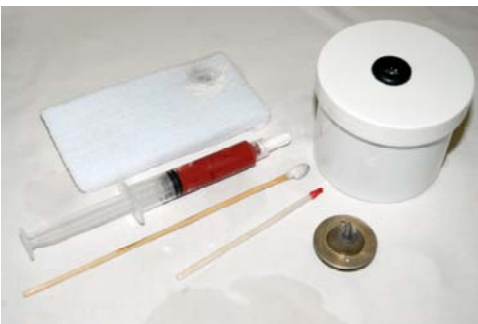
After creating a light paste with fine alumina powder, polish your cone tip using a felt pad or cotton cloth. Though the felt pad is gentle, do not apply excessive pressure directly on the tip of the cone. Certain skimmer cones are particularly prone to blunt force trauma due to their thin walls and sharp edges.



A cotton swab is very effective at cleaning the inside (concave side) of the cone. Using the same alumina paste, clean as close to the orifice as possible. By creating a point at the tip of the cotton swab it will be possible to clean residue from the inside edge of the orifice.



For optimal cleaning of the cone orifice Spectron recommends using diamond paste and a foam tipped cleaning stick. These sticks simplify the effective cleaning of the most important region of the analytical zone.



At left are the tools we recommend for mechanically cleaning your cones. For additional information send us your questions to: [Spectron@SpectronUS.com](mailto:Spectron@SpectronUS.com)

## Appendix C

### Platinum Cone Refurbishing



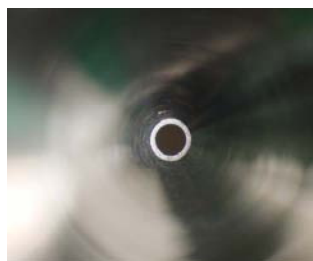
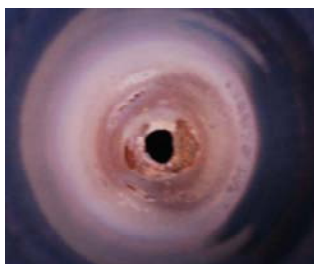
**Platinum cone damaged from mishandling:** Since the metal is in good condition; neither annealed nor chemically degraded, it can be easily refurbished. Our free Pt refurbishing process will reform the cone to within the manufacturer's original specifications.



**Platinum cone chemically degraded beyond repair:** Here the cone has been made brittle and porous. This cone is ready for Pt reclamation. For the current *Pt Reclaim Credit* values go to the **Library and Resources** section of our web site.

### “What goes around comes around”

Free platinum cone refurbishing



Before and after images of a professionally refurbished cone: With proper tools and experience it is possible to return cones to their original OEM specifications.

## **Acknowledgments**

The art of cone cleaning has evolved over the 20 years I have been part of the ICP-MS community. There are no specific sources I can cite for the information described here. Most of it was gained anecdotally and through personal experimentation. I would like to acknowledge however, Agilent Technologies, PerkinElmer and ThermoFisher. My relationship with persons in each of these companies has surely been responsible for some of the knowledge I am passing on to each of you. I would also like to thank Dr. Tom Rettburg of VHG Labs for assisting in the editing of this document.

Lawrence Neufeld/President Spectron, Inc.

## **Available Accessories and Supplies**

### **Cone Cleaning Kit (SI-CCK01)**

- Fine Alumina Powder
- Diamond Abrasive Paste
- Thick Polyester Polishing Felt
- Cotton Swabs
- Conical-Tipped Foam Swabs
- Synthetic Fiber, Lint Free Cloth
- 10X Magnifier Lens

### **Bath Sonicator (SI-E15H)**

- Stainless Steel, cavitation resistant tank
- 5.9 x 5.4 x 3.9 (W x D x H)
- Permanent sweep function for optimized sonic distribution
- Temperature controlled ultrasonic operation

### **Cone Cleaning Fixture (SI-CCFxx1)**

Cleaning fixture for safely clean cone tip

- Sampler = 1 Skimmer = 2
- Agilent xx = AT
- Nu Instruments xx = NU
- PerkinElmer xx = PE
- Thermo Elemental xx = VG
- Thermo Finnigan xx = FN
- Varian xx = VN