

## CLEANING MICROMOUNTS

Most micromounts need to be cleaned at some point during their preparation for mounting. Dirt and other contaminants should be removed to enhance the aesthetic value of the specimen, and yet care must be taken to do this enhancement without damaging the specimen. Care must be taken to avoid removing a coating or encrustation which is itself a significant species and thus a major part of the overall specimen.

These contaminants include material present on the specimen when mined, chips and dust collected during trimming, lint of various kinds from storage materials, and airborne matter accumulated while sitting in an uncovered state. Unwanted materials which are an integral part of the specimen include iron and manganese oxides, hydrocarbon coatings, encrusting minerals, and visually uninteresting parts of the specimen. (Remember that many of these "unwanted" materials are an integral part of the original paragenesis, and thus have scientific value which may justify keeping them on the specimen.) A variety of cleaning techniques are available to deal with these contaminants which include mechanical measures, air, water, and chemical cleaners. The use of an ultrasonic cleaners can enhance many of these techniques..

Wrapping materials are notorious for generating lint which clings to acicular crystals. Avoid the use of jeweler's cotton, toilet and tissue paper, paper towels, and other lint producing materials for wrapping. Tissue paper of the kind for wrapping packages is relatively free of lint, as is newspaper. Colored paper may permanently stain some minerals, particularly zeolites, if it is placed in contact with a damp specimen. Small plastic bags work well. Another good wrapping material is the ultrathin plastic sheeting which comes on clothing from the dry cleaners or Saran Wrap™. This material can be loosely wrapped around specimens or balled up and used as packing inside a mailing box in much the same way that styrofoam pellets are used, and can actually be stuffed inside micromount boxes to provide a shock absorber during shipment of mounted specimens.

A sudden shift of temperature during the cleaning process can cause thermal shock which can crack or fracture some crystals (fluorite is particularly sensitive to sudden temperature shifts). For this reason, cleaning procedures should be performed with all the solutions at the same temperature. If solution temperatures have been raised during the cleaning process, bring the specimen back down to room temperature by letting the first rinse slowly equilibrate with room temperature. If a double boiler arrangement is used during cleaning, the water bath part of the system can be utilized as the first rinse since it will start out at the same temperature as the cleaning solution.

The energy of an ultrasonic cleaner is transformed into heat with subsequent elevation of the solution temperature. Moving a specimen from the elevated temperature of the ultrasonic bath to a room temperature rinse may cause thermal shock. The use of a hair dryer or a warm oven may also cause a rapid change in temperature with resultant damage.

If a diamond saw is used for trimming specimens, be sure to use a water soluble coolant which has little in the way of chemical additives. Any oil based coolant will be extremely difficult to remove from the tiny crevices of micromounts. Ethylene glycol (antifreeze), either straight or somewhat diluted, works very well and is totally water soluble, but it is poisonous if ingested or absorbed through the skin to any degree. Another option is to use plain water and a stainless steel blade, draining the water after each use.

## MECHANICAL CLEANING TECHNIQUES

Mineral species are chemicals, and as such are vulnerable to attack by many of the chemicals used in the cleaning process. Some are affected by water, while others seem to be inert to the actions of almost anything we throw at them. Remember that the degree of reactivity may vary significantly among the various species on a single specimen, and therefore the fewer chemicals (and this includes water) which you have to apply to a specimen the better. Dry techniques are the safest way to start the cleaning process. Human breath is neither dry nor clean. It contains a great deal of moisture which can harm some species and can induce dust to adhere to the specimen. There are also substances in breath which can leave stains or scum on the surface of crystals.

- **Tapping.** Hold the specimen upside down and tap the reverse side of the specimen or the back of the hand or fingers holding the specimen. This will often knock loose material off the specimen.
- **Squeeze bulbs** are generally available through camera stores and are used for cleaning photographic equipment. Ear syringes are available in pharmacies and are also effective. They can be deliver air as a gentle puff or a relatively strong blast. Air blown straight onto the front surface of a specimen can force the dirt deeper into the cavities, while directing the flow parallel to that surface is more likely to remove the dirt. Using the squeeze bulb with the specimen upside down may enhance the cleaning, and if you are really dexterous, tapping at the same time will help to loosen the dirt.
- **Pressurized air** is available in a variety of delivery systems. Some are 100% disposable while others have a disposable canister but a reusable nozzle. The best ones are those developed for cleaning photographic or electronic/computer equipment. They can be used at low pressure if you are careful, but generally they deliver a very strong stream of air. This can be very effective for cleaning, but obviously is capable of removing some of the material which you are trying to clean, particularly acicular or loosely attached crystals. The systems available now are usually ozone friendly.

Pressurized air can also be delivered by using a 25 gauge or smaller hypodermic needle attached to a syringe. The sharp end of the needle makes a very good pick for lifting out lint or digging out other unwanted materials.

- **Vacuum cleaner.** Creating airflow across the surface of a specimen by suction will often remove unwanted dirt with less damage than with positive pressure. Holding the specimen upside down and tapping it at the same time may facilitate the cleaning process. Practice using the vacuum cleaner on unwanted specimens or you may lose your prize specimen down the tube. If there is an exhaust port on the vacuum cleaner which allows the attachment of a hose, you may be able to use it as a source of positive pressure air as well.
- **Brush.** A tiny artist's paint brush (usually camel hair or red sable) which comes to a very fine point can be lightly dampened and used to pick off hairs or other tiny fragments. This will not work if the specimen is already wet. A toothbrush can be used on hard specimens.
- **Forceps, tweezers.** Very fine forceps such as those used for eye surgery or watch repair can be used to remove individual fibers or hairs.

To be continued next issue

(Continued from the Newsletter #189)

## SPECIES SPECIFIC CLEANING TIPS

- *Aurichalcite* and *velvet malachite* are O.K. in an ultrasonic cleaner for a short period of time. (19)
- *Carbonates* can frequently be cleaned using full strength bleach. Sodium dithionate can be used to remove rust stains safely from many carbonates, but since it is a weak acid, test first.. Ascorbic and citric acids are usually safe cleaning solutions but test first.
- *Calcite* can often be dissolved using vinegar with little chance of dissolving other species. **Test first!**
- *Copper (British Museum Method)*. The following formula attacks the black copper oxides but does not attack the copper or the commonly attached cuprite. The solution consists (by weight) of 1 part sodium hydroxide (soda lye), 3 parts rochelle salts (potassium sodium tartrate) and 20 parts H<sub>2</sub>O. Soak time may be a few minutes to one hour.  
  
*Copper (calcite removal)*. A dilute solution of sulfuric acid, 1 part acid by volume to 4 parts H<sub>2</sub>O will effectively remove calcite without seriously affecting the copper.
- *Denture cleaner* has been advocated as a cleaner. Your editor has not tried this, nor does he know the chemistry of the cleaners, so you are on your own. Test with a sample first.
- *Fluorite* is extremely heat sensitive, so be careful with ultrasonic cleaning.
- *Gypsum, variety selenite*, has perfect cleavage on {010} which allows the penetration of water. Use of detergents will result in deposition of a soap films on the cleavage planes leaving a cloudy crystal. These same cleavage planes also make it difficult to completely remove residual acid from the crystals.
- *Hydrocarbon* deposits on the surface of specimens are part of the natural paragenesis, and removal does alter the mineral suite. If removal is desired, ethyl alcohol, Thin-X™, benzene, and carbon tetrachloride have all been used with varying degrees of success. Benzene and carbon tetrachloride are both dangerous chemicals.
- *Phosphates*. Do not use a phosphate-based detergent for cleaning as this may well leave a residue on the specimen. (19)
- *Pyrite, marcasite, and pyrrhotite* should be cleaned in H<sub>2</sub>O if no other cleaning method will remove the foreign material. Marcasite will deteriorate less over the years if water is not used. An overnight soak in vinegar will brighten these minerals as will a soak in dilute oxalic acid.
- *Silver* can be cleaned by electrolysis. Place the specimen in an aluminum container along with a solution containing 1/2 oz NaHCO<sub>3</sub> and 1/2 oz NaCl per quart of solution. The solution can be gently heated to speed up the reaction. (Pearl)
- *Zeolites*. Most zeolites can be safely cleaned in some kind of acid. Strong acids will reduce many of them to a silica gel. The acid reactivity is directly related to the mineral's silica content. The low silica zeolites react much more quickly to acid than the high silica ones do. The very high silica zeolites like mordenite are very resistant to acids. (8)

## MECHANICAL CLEANING TECHNIQUES (continued from last issue)

- **Needles and other crow bars.** Sewing needles come in a great variety of lengths and calibers. They can be used to loosen attached fragments of dirt. Moistening the needle by touching it to the tip of your tongue can facilitate the removal of stubborn fibers. Some needles are long enough to hold with your fingers, while others require a pin vise or handle of some type to be useful (a 1/4" dowel with an appropriately sized hole makes a good handle). The stiffer ones, particularly sewing machine needles are strong enough to pop off unwanted or broken crystals or pieces of matrix. For ultrafine work, fine gauge hypodermic needles with their extremely sharp points are invaluable. If the hypodermic needle is attached to a syringe, an air stream can be directed at the same time. Dental picks may be helpful when used for cleaning debris from hand specimens, but under the microscope they have all the finesse of a crow bar.
- **Bamboo skewers.** Bamboo is quite soft and yet can be split into fine slivers to use as a pick. The sliver can be moistened and used to polish the surface of micro crystals. The skewers can be obtained from novelty or party shops.
- **Static electricity.** Rubbing a pair of fine tweezers or a needle with a piece of nylon or wool can create a static electric charge on the tool. This is often strong enough to pick off tiny fibers or lint.
- **Cement bead.** Some collectors advocate using a tiny bead of tacky material such as contact cement (before it dries completely) to lift off dirt. This bead is placed on the end of a fine needle or other tool and then touched against the particle which you want to remove in much the same way as the damp brush is used. A tiny residue of Mineral Tack™ or similar adhesive putty on the tip of a needle can also be effective.
- **Balsa stick.** A 1/16 inch balsa stick, cut twice on a diagonal to give a soft point, is very effective in polishing crystal faces and removing soft scum.

## CLEANING WITH WATER

Water is not necessarily pure H<sub>2</sub>O. Tap water may contain chlorine, calcium and magnesium ions, and other chemicals. Rain water may contain dissolved gases which can produce slight acidity. Many of these ions are capable of reacting with other cleaning agents as well as with the specimen. The alterations on a crystal face caused by a mild acid or alkali reaction may not be obvious on a hand specimen, but when the specimen is viewed through the microscope and you see crystal faces pitted or covered with a scum from your tap water, do not be surprised. If cleaning has to be done with tap water, a good rinsing at the end of the cleaning process with distilled water is recommended. Distilled water is available in the supermarket by the gallon. The runoff from your dehumidifier is essentially distilled water and is free. Deionized water can be purchased in the grocery store. A deionizing kit can also be purchased in a grocery store. It is a simple ion exchange system. In the following discussions "H<sub>2</sub>O" will refer to distilled water.

Water works best as a cleaner when warm, and particularly when used in conjunction with detergents. Rapid immersion of a specimen into very warm or hot water can precipitate thermal shock.

- **Thermal shock.** When using any liquid-based cleaning method be aware that sudden changes in temperature can result in rapid expansion or contraction of crystals with subsequent cracking or breaking. If the cleaning process is started at room temperature then it is relatively easy to allow all specimens to return slowly to room temperature and thus prevent cracking or breakage.

- ***Water soluble species.*** As a general rule, nitrates, borates, chlorides, sulfates and a few carbonates are soluble in H<sub>2</sub>O to a greater or lesser degree. The solubility of marginally soluble species such as malachite and azurite rises rapidly with increases in solution temperature. Extended use of the ultrasonic cleaner will increase the temperature of the bath. Minerals which form in brine such as hanksite and halite should be washed in the brine solution at the time of collection or washed in 95% alcohol.
- ***Clay family minerals*** may be classed as insoluble, but they can absorb great quantities of water with subsequent fragmentation and disintegration.
- ***Micas*** can split along cleavage planes from immersion in water.
- Minerals with “*meta*” in their names may well be altered by submersion in water through the process of rehydration.
- ***Minerals which tend to oxidize***, as many sulfides do, should probably not be washed in water. Alcohol is usually a safe, although not very effective cleaning solution.
- ***Acicular minerals***, particularly those which form mats, will tend to clump together irreversibly when immersed in water. Optimal cleaning is one of the “dry” techniques, but sometimes the use of alcohol as a cleaning agent will work. Try it on a discard first. Very occasionally the use of a wetting agent or surfactant (such as detergent) will prevent the clumping when using water.
- ***Residual water*** left after cleaning can damage the specimen or cause the deposition of a scum on crystal faces. Excess water can be removed by placing the specimen on a lint-free piece of dry cloth or newspaper, preferably face down if the crystal structure will permit. A stream of air can be passed tangentially over the specimen to both blow off excess water and to speed up evaporation. A hair dryer can be used but watch out for thermal effects. The specimen can also be rinsed in alcohol to remove the water.
- ***Soaking*** may loosen adherent matter to the point where it can easily be rinsed off or picked away using fine bamboo slivers or a needle.
- ***Agitation.*** Suspend the specimen upside down in water and gently agitate.
- ***Running water.*** Dripping water may be all that very delicate specimens can tolerate, while sturdier specimens may tolerate the full force of tap water. Use room temperature water since significant temperature changes can cause thermal fracturing.
- ***Pulse jet.*** This irrigator is the sort used for massaging gums and cleaning teeth, but it does produce a fairly strong water jet. It is sold in pharmacies under the name Water Pik™.
- ***Pressurized water.*** Water can be put under a fair amount of pressure using a syringe and small hypodermic needle. A pinhole attachment for a garden hose can cause a very fine but strong jet of water.

- **Ultrasonic cleaner.** The use of an ultrasonic cleaner is not particularly complicated, but there are limitations to what it can do. There is an optimal filling level of the tank on each cleaner, so read the instructions.

a) The energy generated by the cleaner is capable of disintegrating specimens, so tend to use short cleaning times. Most cleaning can be done in just a couple of minutes. Prolonged cleaning can increase the risk of specimen damage as well as cleaner damage, and can significantly raise solution temperature.

b) If the dirt is packed on the specimen consider a presoak. Depending on the material to be cleaned, wetting agents, detergent, ammonia, weak acids, etc. can be added to the bath. Verify that the agent being added will not damage the specimen or the tank. If chemicals such as acids are used which might damage the tank, make a double bath using a glass container to hold the specimens and cleaning fluid and suspend this off the floor of the tank. One author (4) suggests not using plastic containers since the sound waves may be absorbed by the plastic, reducing the effectiveness of the cleaner. Many of us have used plastic containers or supports in an ultrasonic cleaner without obvious loss of cleaning power. The tank should be filled with water to the appropriate level.

c) Keep specimens and fingers off the floor of the tank. They can dampen the action of the cleaner if they are resting on the bottom, and can actually damage the transducer which is attached to the undersurface of the tank. Use the specimen basket provided by the manufacturer or make one which is suspended above the floor of the tank. A wire mesh basket hung from the sides of the container works well. Sound waves are transmitted by glass.

d) The energy generated by the cleaner can damage you as well as your specimen. If you are in the same room as the cleaner, use of ear protection is recommended by some authors even though you cannot hear the sound although most people think this is generally not a problem. Cleansing of fingers can result in skin damage much like a burn, and can cause pain in the joint capsules.

e) Since the dirt is often loosened rather than removed, rinse specimens well with distilled water at the end of cleaning. If the specimen will tolerate the position, a second cleaning in fresh distilled water with the specimen upside down on a soft bed of material can remove a lot more dirt.

f) Placing the specimen upside down in the ultrasonic cleaner may allow tiny particles which otherwise might have become lodged in cracks to fall off the specimen. Make sure that the good side of the specimen rests on something soft such as a piece of cloth or sponge.

g) Many of the chemicals and compounds used for cleaning specimens are capable of corroding metals including stainless steel, so it is always a good idea to do cleaning in a glass container which has been placed inside the cleaning tank of the ultrasonic cleaner.

h) The ultrasonic cleaner is capable of rounding the edges of sharp crystals of gold and other soft elemental minerals. (19)

## PRECAUTIONS FOR CHEMICAL CLEANING

- Assume that all chemical cleaning products are hazardous to both the specimen and yourself. Follow the guidelines listed below regarding personal protection, and pretest an spare sample before your good specimen is bubbled away.
- Wear goggles whenever using acids, alkalis, or caustic materials. Even one drop of a chemical in the eye can cause permanent damage. Good quality rubber gloves are also advisable.
- Make sure that you know exactly what chemical you are using, as well as its concentration and effects on human tissue. Have a neutralizing agent on hand at all times.
- Keep a good supply of household baking soda or powdered garden limestone on hand to neutralize acid spills. Apply liberally and then flush with water.
- Keep a large bottle of white vinegar on hand to neutralize alkali spills. Apply liberally and then flush with water.
- Always have plenty of water available for immediate flushing and dilution of spills.
- Store chemicals out of the reach of children.
- Keep all containers clearly labeled with the name of the chemical and its concentration. If the material is a combination of chemicals, add a mixing date to help determine if it is still effective.
- Never pour water into an acid. Pour a thin stream of concentrated acid into cold water. The mixing of acid and water generates heat to the point where the water will boil and splatter if added directly to the acid. This is most likely to happen with sulfuric acid.
- Some chemicals such as nitric acid generate toxic fumes when open to the air. Others give off fumes as a by-product of the cleaning process. Have good ventilation available or work outside (upwind) under these circumstances.
- Do not store or use acids near tools as they will quickly rust. This is caused by the acid fumes, not just liquid acid, so your microscope, light, etc. are all at risk.

## CLEANING SOLUTIONS (GENERAL)

Micromounts often contain multiple species, many of which are important to save, either to document paragenesis, or simply for aesthetic reasons. Since each of the cleaning solutions listed in the next few sections may react very differently with different species, it is important to look at the whole mount, not just a particular species before deciding on a cleaning solution. As a general rule of thumb, soak a specimen which has been cleaned with some substance other than distilled water, for twice as long as the cleaning soak to remove all traces of the cleaner. Residual traces of the cleaning agent can cause discoloration and continued chemical activity so do not cut short the final rinse.

Prior to cleaning a specimen with a chemical bath, it should be soaked in water containing either a detergent or a surfactant for about 20 minutes. This will allow the water to replace air pockets on the specimen and facilitate the penetration of the cleaner into all cracks and crevices.

- **Acetone** ( $\text{CH}_2\text{COOH}_3$ ). This is an alternative to alcohol for water soluble species. It will remove lacquer and many adhesives. It is very flammable to the point of easily creating an explosive atmosphere, and the fumes are toxic. No neutralizer is necessary. Its rare use does not justify keeping it in a home laboratory.
- **Alcohol, ethyl.** Water soluble minerals can usually be safely rinsed in alcohol. It can also be used to rinse acicular minerals which would tend to mat together when rinsed in water (test a spare specimen first). Alcohol can be used to remove residual water from specimens after a water wash to prevent spotting and to speed up the drying process. Alcohol will dissolve shellac and many forms of grease including fingerprints. Ethyl alcohol can be obtained as 190 proof (95%) denatured alcohol at paint stores. It is flammable (but less flammable than acetone) and in its denatured form it is toxic if taken internally. The denaturing additives are used to make it nonpalatable but are generally not harmful to mineral specimens.
- **Alcohol, methyl.** This alcohol will work just as well as ethyl alcohol, but it is extremely toxic to most living tissues including humans. This is commercially available as 95% alcohol by weight. It is more flammable than ethyl alcohol. There is no good reason to keep this in the home laboratory purposes if ethyl alcohol is available.
- **Alcohol, isopropyl.** This alcohol can also be used for cleaning or drying, but check for the presence of additives such as perfumes when getting it from a drugstore, as they can leave stains and scum on mineral specimens. It can be obtained in hardware stores and is less flammable than ethyl alcohol. .
- **Alconox** is a wetting agent (surfactant) which can be added to presoaks to help "wet" the specimen prior to chemical cleaning or use of the ultrasonic cleaner.(19)
- **Ammonia** ( $\text{NH}_4$ ). "Household ammonia" commonly contains various additives such as detergents which may make it unsuitable for use as a reagent. "Clear" ammonia is available at the grocery store. Some mineral species will chemically react with ammonia. Lichens and algae can usually be loosened up with a prolonged dilute ammonia soak (also in chlorine bleach). Lichens secrete acids which are capable of slowly eroding the surface of specimens and thus such specimens may be dull or etched under the microscope. Soaking specimens which have a coating of clay in a weak ammonia solution can often loosen up the clay enough to allow its removal with an ultrasonic cleaner or running water. Concentrated ammonia can damage skin, eyes, and mucus membranes. It is neutralized by vinegar. Do not use this on turquoise as it turns the turquoise white.
- **Benzene and carbon tetrachloride** can be used for removal of hydrocarbon deposits but both are toxic and not recommended for stocking in the micromounter's laboratory. If a hydrocarbon solvent is needed, ones such as Thin-X™ are relatively safe and probably work just as well. (Thin-X™ is good for taking old gum from labels off plastic boxes without damaging the plastic.)
- Biz™ bleach (a mixture of enzymes, sodium perborate bleach, water softeners and sodium phosphates) has been recommended for cleaning zeolites. Carefully test other species which have significant solubilities.

- **Chlorine bleach** (NaOCl, Javel water, Javex, Chlorox™) is useful for the removal of green algae that is found on some of the zeolites from Nova Scotia. A 10 to 25% solution of Chlorox™ (5.25% NaOCl) in H<sub>2</sub>O usually works well. It may take anywhere from a few hours to a couple of days depending on the amount of material to be loosened. Using an ultrasonic cleaner while soaking can significantly speed up the process. Acid sensitive minerals such as carbonates and phosphates can often be cleaned with bleach. Use a glass insert to protect the tank. Full strength chlorine bleach is often very effective in cleaning carbonate minerals. When mixed with acid (including vinegar!) it will release extremely irritating chlorine gas. Never mix with ammonia as extremely toxic gases will be released.

The following is from "Geminews" of the Montreal Gem & Mineral Club, July 1985. "Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) 15% etches like Javel water. It also reacts violently and produces oxygen when mixed with Javel water. This property can be successfully applied to effectively clean minerals with relatively little risk. First place your mineral in Javel water for about 24 hours to soak the sand and clay deposits. Then submerge it in hydrogen peroxide (careful) and because of the violent reaction the sand and clay particles will be *blown* off the specimen. This process can be repeated until the mineral is clean." "The above article is based on a publication which appeared in the Journal of the Netherlands Lapidary Club. It is offered for your careful consideration or use, but neither the editors nor the Montreal Gem & Mineral Club can be held responsible for mishaps of any kind"

- **Coca-Cola™** (and other carbonated drinks) has been touted for removal of kaolin-type encrustations on pegmatite minerals. Soak the specimen in Coca-Cola™ for 24 hours and then remove material manually or with an ultrasonic cleaner. Your editor has no experience with this technique. Perhaps carbonic or citric acid is the cleaning agent. (18)
- **Detergents.** If detergent is used, the liquid variety is preferable since the gel formed by incomplete dissolution of particles can be very difficult to remove from specimens. Some detergents contain chlorine additives which will react with ammonia to release chlorine gas, so make sure you read the labels carefully. Many detergents are highly fluorescent, and a porous specimen may retain enough detergent to take on an artificial fluorescence.
- **EDTA** (*versene, ethylene diamine tetra-acetic acid, ethylene dinitrilo-tetra-acetic acid*) is a chelating agent which can be added to acid baths in ratio of 1:4, EDTA to acid by weight. This will entrap metal ions, particularly those that are dissolved by acid cleaning, and prevent those ions from staining areas around cracks and fissures. The EDTA may react with some minerals, so test first. The chelating reaction is also pH dependent, becoming less effective the higher the acid concentration.
- **Hydrogen peroxide** (H<sub>2</sub>O<sub>2</sub>) can reportedly be used in the same manner chlorine bleach particularly with minerals which are acid sensitive. It is a reasonably strong oxidant so preliminary testing is probably advisable. See comments under chlorine bleach.
- **Sodium hydroxide** (NaOH). This is available at the grocery store as caustic or washing soda, and also as drain cleaner. Neutralize with vinegar then use a prolonged H<sub>2</sub>O rinse.

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## CONTINUED FROM LAST NEWSLETTER

- **Sodium sulfite** ( $\text{Na}_2\text{SO}_3$ ), **sodium metabisulfite (AKA sodium pyrosulfite)** ( $\text{Na}_2\text{S}_2\text{O}_5$ ). Any one of these is useful for removal of manganese oxides. A hard clay coating which will not come off with normal cleaning techniques can often be removed by a gentle boiling in a strong solution of sodium sulfite (one part sodium sulfite to four parts  $\text{H}_2\text{O}$  by weight). It is used as a concentrated solution and is a weak acid. Skin contact is usually not a problem with prompt water washing, but it does generate sulfur dioxide which is a very toxic gas, and therefore it should be used with good ventilation present. Care should be taken with minerals such as carbonates and phosphates which are acid sensitive. These materials are sold in hardware stores as Iron Out™ (Ace Hardware) and Power Rust Remover™. The use of Pyrex™ containers is recommended because the acid may etch metal containers.
- **Soft Scrub™** is a cleaner which has calcium carbonate particles suspended in a cleaning solution. It has been used with a tooth brush for removing hydrocarbon scale from serandite. Some varieties of it contain bleach. Has anyone tried this as a dilute solution in an ultrasonic cleaner?
- **Spray/foaming cleaners** (such as Bon Ami™) are reported to work well for removing adherent dirt. Spray the substance on the specimen, let it soak for a few minutes, and then wash it well, using an ultrasonic cleaner if necessary. (Ark. Rock News) Your editor has no personal experience with this technique.

## REMOVAL OF OXIDES AND HYDROXIDES

Most techniques for rust removal depend upon the increased solubility of ferric oxides in acidic solutions and the subsequent sequestration of the ferric ions by another anion. Attempts to terminate the process with alkali will result in the formation of ferric hydroxide which will leave deposits on the specimen. These are often harder to remove than the original rust deposits. Use copious and prolonged  $\text{H}_2\text{O}$  rinses at the end of the cleaning process for termination of the chemical reaction.

If a rust cleaning solution starts becoming dark, the specimen should promptly be removed and placed in fresh solution because the now dissolved iron or other coloring agents can work their way into cracks and crevices where they can produce new stains.

- **Ascorbic acid** (Vitamin C). This serves as a chelating agent which solubilizes metal cations. Use approximately 1/2 tablespoon of powder in a cup of warm  $\text{H}_2\text{O}$ . This will remove most *manganese* oxides in less than a minute. Removal of stubborn stains can be enhanced by the use of the ultrasonic cleaner. Make only as much solution as you plan on using because it will turn yellow and lose its effectiveness in about 12 hours. If the powdered form is not available, vitamin C tablets can be ground up to make the solution.
- **Hydrochloric acid** in a dilute form will often remove manganese oxide coatings.
- **Oxalic Acid** ( $\text{COOHCOOH}\cdot 2\text{H}_2\text{O}$ ). This is another chelating agent. It is strongly recommended that distilled  $\text{H}_2\text{O}$  be used for making oxalic acid solutions because calcium will be precipitated from tap water as insoluble oxalates. A solution of 1 cup of oxalic acid crystals per gallon of  $\text{H}_2\text{O}$  can be used to chemically remove *iron* stains. If simple soaking does not work, heating the solution to just under the boiling point for about 1/2 hour may help. Some collectors have advocated actual boiling of the specimens for up to 2 hours in an oxalic acid solution. Do not permit the specimen to stay in the bath as it cools because the fluid may be drawn into pores in the specimen with later weeping, encrustation, and staining. Move the specimen into a bath of distilled  $\text{H}_2\text{O}$  of approximately the same temperature, changing the  $\text{H}_2\text{O}$  frequently. Others have recommended a saturated solution of oxalic acid rather than the somewhat dilute solution recommended above. The saturated solution can be made by adding oxalate crystals to hot  $\text{H}_2\text{O}$  until no more crystals will dissolve. As the solution is brought down to room temperature, crystals will form in the container indicating that it is saturated. Do not use metal containers with oxalic acid as it will etch the metal. A tap water rinse after oxalic acid treatment will precipitate insoluble oxalates on the specimen.

Oxalic acid, as its name states, is an acid, and thus can react chemically with many species especially carbonates and phosphates. If any question exists about the potential for acid reaction with the specimen, test a disposable specimen first. Occasionally you will end up with a pseudomorph of some oxalate after the desired species. Failure to totally remove all traces of the acid will allow continued leaching and discoloration, particularly with iron bearing minerals, and may even result in the deposition of insoluble oxalates in cracks and crevices. If the cleaning solution becomes yellow, exchange it promptly for a fresh solution since the dissolved iron may precipitate out as stains of insoluble oxalate on the specimen.

Oxalic acid will generally not damage clothing, and minimal skin exposure with dilute solutions is generally not a problem although a burn can occur with concentrated solutions. Taken internally it is a poison. Oxalic acid is capable of corroding stainless steel, so any cleaning should be done in a glass container inserted into the cleaner tank.

- **Sodium sulfite** ( $\text{Na}_2\text{SO}_3$ ), **sodium metabisulfite (AKA sodium pyrosulfite)** ( $\text{Na}_2\text{S}_2\text{O}_5$ ). Any one of these is useful for removal of manganese oxides. A hard clay coating which will not come off with normal cleaning techniques can often be removed by a gentle boiling in a strong solution of sodium sulfite (one part sodium sulfite to four parts  $\text{H}_2\text{O}$  by weight). It is used as a concentrated solution and is a weak acid. Skin contact is usually not a problem with prompt water washing, but it does generate sulfur dioxide which is a very toxic gas, and therefore it should be used with good ventilation present. Care should be taken with minerals such as carbonates and phosphates which are acid sensitive. These materials are sold in hardware stores as Iron Out™ (Ace Hardware) and Power Rust Remover™. The use of Pyrex™ containers is recommended because the acid may etch metal containers.
- **Sodium dithionate** ( $\text{Na}_2\text{S}_2\text{O}_6 \cdot 2\text{H}_2\text{O}$ ) (method of Robert Waller). This technique works at an optimal pH of 7.3, and therefore is ideal for removing rust stains from acid sensitive species such as carbonates and phosphates. Make up a stock solution consisting of 71 gm sodium citrate and 8.5 gm sodium bicarbonate in one liter of  $\text{H}_2\text{O}$  (this is a stable solution). Place the specimen to be cleaned in the smallest container possible (a plastic bag may be used) and add a premeasured volume of stock solution, enough to cover the specimen. Add 1 gm sodium dithionate to each 50 ml of stock solution. Each 50 ml of solution is capable of removing up to 0.5 gm of ferric oxide. If small volumes are adequate, as for a couple of micromounts, the stock solution may be diluted as a 2:1 ratio of  $\text{H}_2\text{O}$  to stock solution (still add 1 gm of sodium dithionate). Further dilution reduces the efficacy of the method. The dithionate containing solution will remain active for about 12 hours, but most rust removal will be accomplished in 3 to 4 hours. If stains persist at the end of 12 hours, fresh solution must be used. The cleaned specimens should be rinsed in distilled  $\text{H}_2\text{O}$  for at least 36 hours, with water changes every 12 hours. The cleaning process can be speeded up by performing several washes in an ultrasonic cleaner using  $\text{H}_2\text{O}$  (deionized is even better). The used solution may be washed down the drain since it is not ecologically harmful and it will not damage pipes.

The sodium dithionate reduces ferric iron to ferrous iron. The sodium citrate sequesters the ferrous ion, and the sodium bicarbonate buffers the solution. Sodium dithionate is broken down by the humidity in the air, and thus the lid must be sealed carefully after each use. Citrate will also sequester calcium ions, so slightly soluble calcium minerals such as calcite and messelite may be dissolved or damaged by the process. *Test first!*

- **Sodium dithionate** ( $\text{Na}_2\text{S}_2\text{O}_6 \cdot 2\text{H}_2\text{O}$ ) (Mogens Hansen's variation on the method of Waller). Because of the unpleasant sulfur-containing fumes generated by the Waller method and because minerals containing heavy metals with dark sulfides (e.g. lead) could become contaminated on the crystal surface with a grayish coating, the following modification of Waller's method was developed. The stock solutions consists of 59 gm sodium citrate and 28 gm sodium bicarbonate in 1000 cc deionized  $\text{H}_2\text{O}$ . For each 30 ml of stock solution used, 1 gm sodium dithionate is sprinkled over its surface. After 5 minutes, another 1 gm is added in the same manner. The specimen is left at room temperature for 24 hours and then the solution is discarded. The specimen is first washed with a little buffer solution and then several times with  $\text{H}_2\text{O}$ , preferably using an ultrasonic cleaner. Because many unwanted coatings on minerals contain iron (not just oxides and hydroxides) many types of coatings can be successfully removed with this method leaving lustrous surfaces.

## CLEANING SOLUTIONS (ACID)

The reactivity of acids is dependent on the concentration of acid, pH, particle size and temperature. The rate of reaction rises rapidly with an increase in temperature and decrease in particle size. Room temperature or an unspecified temperature is assumed to be approximately 72°F. Solutions may be kept chilled in a refrigerator if a cold reaction is wanted. The temperature can be raised by the application of heat. Sometimes placing an incandescent bulb close to the solution is all that is needed to raise the temperature of the solution enough to speed up the reaction. The energy generated by the cleaner will rapidly increase the temperature of the solution. Raising the temperature of the solution may also increase its reactivity with the container, so if an ultrasonic cleaner with a stainless steel tank is being used, a glass bowl (Pyrex™ is best) should be used like a double boiler to hold the solution whenever chemicals are involved. Any double boiler, whether for use in the ultrasonic cleaner or on the stove, should have the inner glass container elevated off the bottom by a minimum of 25 mm.

Many times dilute acid will do just as good a job as a more concentrated solution with less effect on other areas of the specimen. Start with dilute solutions and room temperature or a lower temperature particularly with species which have a tendency to react to acid.

Dropping very reactive minerals such as calcite directly into strong HCl can cause widespread spattering of the acid or even rupture of the vessel because of the sudden generation of heat. Because of the release of CO<sub>2</sub>, a mineral and an acid should never be placed together in a tightly closed container. When diluting acid always add the acid to water, to avoid splatter of the acid.

Acids are particularly useful for removing calcite or other carbonate overgrowths from other minerals which are acid resistant. With some carbonates other than calcite, the process can be slow. This may be speeded up by either gently heating the solution or using the energy from an ultrasonic cleaner for a few minutes. Alternatively, everything not to be etched can be coated with paraffin. This can be removed later by melting or dissolution in paint thinner.

A single crystal in calcite matrix may be exposed by digging a well around the crystal with a pick and then putting drops of HCl or vinegar into the well until the crystal is exposed.

All acids can work their way into cracks and cleavage planes. Failure to adequately rinse all traces of the acid away with distilled water can leave an ongoing reaction. Etching, stains, and deposition of salts, particularly insoluble oxalates can occur. Cleaning in H<sub>2</sub>O using the ultrasonic cleaner will speed up the removal of the acid. Do not try to neutralize the acid with alkali as this may result in the deposition of insoluble silica gels and other materials.

To minimize further reactions thoroughly flush all specimens cleaned in acid with copious amounts of water and then neutralize with a dilute solution of alkali.

Many acids (particularly HCl, HNO<sub>3</sub>, and acetic acid) are volatile. They can be very irritating to both the eyes and nasal passages, and may damage microscope optics.

- *Ascorbic acid* (vitamin C). Discussed under "Removal of oxides and hydroxides."
- *Acetic acid* (CH<sub>3</sub>COOH). Vinegar is about 5 % acetic acid but may contain some additives (use white vinegar). Acetic acid can be used as a mild acid substitute for HCl and is stronger than citric acid. Glacial acetic acid (28%) diluted 1:1 with H<sub>2</sub>O is much more effective than vinegar, particularly when warmed up, and there is no heat hazard with its dilution. If the cleaning solution starts to turn

yellow, exchange it for fresh solution to prevent the precipitation of stains on the specimen. It can cause serious burns, irritation of the mucus membranes, and eye damage. Glacial acetic acid can be obtained from chemical or photographic supply houses. Sodium bicarbonate is the neutralizer. Vinegar is particularly useful for the removal of calcite from a specimen when other species are present which might be damaged by stronger acids.

- **Citric acid** ( $C_6H_8O_7$ ). This is a weak acid which is usually available through a pharmacy. It is particularly useful for etching away calcite when the mineral to be etched out is also acid sensitive, but to a lesser degree than the calcite. Neutralize with water.
- **Hydrochloric acid** (Muriatic acid, HCl). Most etching work, particularly with small specimens, can be done with commercial grade muriatic acid (about 35% by weight), which is usually available through swimming pool suppliers or paint stores. Reagent grade is 36% acid. Do not cover the container where the reaction is taking place tightly because of the generation of  $CO_2$ . Cover the container loosely with a sheet of glass to prevent splashing. Complete removal of all acid at the end of the cleaning process is very important to prevent further leaching and stains. The acid can slowly permeate through plastic containers as fumes unless they are coated with an impervious lacquer finish (not always done). These fumes can attack metals in the vicinity *including your microscope or light source*. The acid should be transferred to glass containers whenever possible. It can be stored at room temperature in a polyethylene container. Sodium bicarbonate is used to neutralize it.

An oxalic acid rinse (10%) can remove the iron stains left after an initial cleaning with HCl. Follow this with an extended rinse in distilled water.

- **Nitric acid** ( $HNO_3$ ) can dissolve some materials which HCl cannot. The same precautions should be taken with it as with HCl, and, in addition it releases the reddish vapors of nitrogen oxide which are very toxic. Nitric acid is such a powerful oxidant that it can cause spontaneous combustion if spilled on finely divided organic material. Nitric acid will decompose on exposure to sunlight, so it needs to be stored in a brown glass bottle or in the dark. There is little reason to keep this in the home laboratory for the cleaning of micromounts since HCl will almost always do the job as well and is much less dangerous. Neutralize with sodium bicarbonate.
- **Oxalic acid** ( $COOHCOOH \cdot 2H_2O$ ) Discussed under "Removal of oxides and hydroxides."
- **Sulfuric acid** ( $H_2SO_4$ ). This is the most dangerous acid to dilute because of heat generation and secondary splatter. In concentrated form it will absorb as much as 100% of its own volume of water from the atmosphere, causing containers of acid to overflow. Like nitric acid, this acid has little value over HCl to the micromounter because of its dangerous characteristics.
- **Aqua regia** and **hydrofluoric acid**. These two acids will often clean up silica encrustations, but they are so dangerous and limited in their use that they are not recommended as stock items for the micromounter.