

AQUEOUS CRITICAL CLEANING: A WHITE PAPER

Cleaning and Monitoring Procedures

Optimizing Your Critical Cleaning Processes Begins With Using the Right Procedures — Then Monitoring Performance to Ensure Results

Getting the best results from any cleaner requires proper mixing and cleaning procedures. This whitepaper includes mixing directions for each type of Alconox, Inc. aqueous cleaning detergent. It also describes methods for monitoring cleaner performance.

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In any industrial cleaning application, “clean” means clean enough to avoid later problems. The key questions are:

- What will happen if the surface isn’t clean?
- How can you ensure that cleaning standards are maintained?

Whether or not cleaning validation is mandated in your operation, it is useful to understand the testing methods commonly used for determining cleanliness of industrial, medical, pharmaceutical, food and other types of processing equipment.

1. Mixing Directions for Alconox, Inc. Detergents

Dilute detergent using warm (about 120°F or 50°C) or hot (about 140°F or 60°C) water per **Table 1**. Ambient temperature water may be acceptable,

especially for presoak. For difficult soils, use very hot water (above 150°F or 65°C) and double the recommended amount of detergent. When cleaning solution may be reused, make up fresh solutions frequently, as needed.

2. Soaking

Recommended Products: ALCONOX, TERGAZYME, LIQUINOX, CITRANOX, DETERGENT 8, LUMINOX, ALCOJET, DETOJET, CITRAJET, TERGAJET, SOLUJET, DETONOX, KEYLAJET

Typical Uses:

- Cleaning small items (e.g., hospital catheters and tubes, small metal parts) and large tank interiors, including pharmaceutical and other blending tanks



In any industrial cleaning application, “clean” means clean enough to avoid later problems. The key question is, What will happen if the surface isn’t clean?

- Pre-treatment to loosen soils and prevent drying — especially for labware or medical instruments prior to further cleaning

Advantages: Very little physical effort or expense.

Concerns: Extremely dirty articles or difficult soils may require further cleaning.

Directions: Soak completely submerged in solution until clean. This may take several hours, depending on the type of soil. Remove and rinse thoroughly (see Rinsing).

3. Manual Cleaning

Recommended Products: ALCONOX, TERGAZYME, LIQUINOX, CITRANOX, DETERGENT 8, LUMINOX, ALCOJET, DETOJET, CITRAJET, TERGAJET, SOLUJET, DETONOX, KEYLAJET

Typical Use: Cleaning small articles such as medical examination instruments, labware or circuit boards, and large articles such as process equipment.

Advantages: Versatile, inexpensive, effective.

Concerns: Time consuming and labor intensive. May not be effective on hard-to-reach areas requiring pre-soak, ultrasonic or machine cleaning.

Directions: Make up cleaning solution per mixing directions, or use undiluted detergent on a warm wet cloth or sponge for non-abrasive scouring. Clean as follows: Wet the article with solution by dunking or using a soaked cloth or sponge. Clean with a cloth, sponge, cotton swab, brush or pad that agitates surface soils without marring the surface. Rinse thoroughly (see Rinsing). Wear gloves, eye protection and other safety equipment if recommended.

4. Ultrasonic Cleaning

Recommended Products: ALCONOX, TERGAZYME, LIQUINOX, CITRANOX, DETERGENT 8, LUMINOX, ALCOJET, DETOJET, CITRAJET, TERGAJET, SOLUJET, DETONOX, KEYLAJET

Typical Use: Cleaning large batches of articles or for fast, convenient cleaning.

Advantages: Fast, effective, penetrating cleaning.

Concerns: Capital cost; material tolerance for ultrasonic agitation.

Directions: Make up detergent solution in a separate container. Add cleaning solution, run machine for several minutes to degas solution and allow heater to come up to temperature. Place groups

TABLE 1: DILUTION INSTRUCTIONS FOR ALCONOX, INC. DETERGENTS

Product	Form	Dilution (%)	Recommended Amount: A: oz/gal, B: gram/l, C: ml/l	Min Wash Temp	Usual Wash Temp
ALCONOX	powder	1	A: 1 1/4, B: 10	Ambient	Warm
TERGAZYME	powder	1	A: 1 1/4, B: 10	Ambient	Max 130°F
LIQUINOX	liquid	1	A: 1 1/4, C: 10	Ambient	Warm
CITRANOX	liquid	1–2	A: 1–3, C: 10–20	Ambient	Hot
DETERGENT 8	liquid	2–5	A: 2–6, C: 20–50	Ambient	Hot
LUMINOX	liquid	2–5	A: 2–6, C: 20–50	Ambient	Hot
ALCOJET	powder	1/2–1	A: 1/2–1 1/4, B: 5–10	Warm	Hot
DETOJET	liquid	1/2–1	A: 1/2–1, C: 5–10	Ambient	Hot
ALCOTABS	tablet	–	(1 tablet per use)	Ambient	Ambient
CITRAJET	liquid	1–2	A: 1–3, C: 10–20	Ambient	Hot
TERGAJET	powder	1/2–1	A: 1/2–1 1/4, B: 5–10	Warm	Hot
SOLUJET	liquid	1/2–1	A: 1/2–1 1/4, B: 5–10	Ambient	Hot
DETONOX	liquid	1–5	A: 1–4, B: 10–30	Ambient	Hot
KEYLAJET	liquid	5	A: 1–6, C: 10–50	Ambient	Hot

Directions: Dilute detergent (see chart) using warm (about 120° F or 50° C) or hot (about 140° F or 60° C) water. Ambient temperature water may be acceptable, especially for pre-soak. For difficult soils, use very hot water (above 150° F or 65° C) and double the recommended amount of detergent. When cleaning solution may be reused, make up fresh solutions frequently, as needed.



Don't neglect the rinse! Use ambient, warm, or hot water. A running water rinse directly contacting all surfaces for at least 10 seconds on each surface is desirable.

of small articles in racks or baskets. Align irregularly shaped articles so the long axis of any part faces the ultrasonic transducer (usually the bottom). Immerse articles to be cleaned for 2–10 minutes or longer, as needed. Remove and rinse thoroughly (see Rinsing).

5. Clean-in-Place

Recommended Products: ALCONOX, TERGAZYME, LIQUINOX, CITRANOX, DETERGENT 8, LUMINOX, ALCOJET, DETOJET, CITRAJET, TERGAJET, SOLUJET, DETONOX, KEYLAJET

Typical Use: Pipe, tank and filtration systems.

Advantages: Assures clean systems without disassembly.

Concerns: Good circulation in system.

Directions: Make up cleaning solution per mixing directions. Circulate solution slowly for at least 1/2 hour. Allow several hours for large systems (1000s of gallons), especially with ambient temperature water. Drain by pumping in one full system capacity of water. Rinse by circulating and draining at least two times the system's water capacity. Some filtration units may require more rinsing.

6. Machine Washers

Recommended Products: DETERGENT 8, ALCOJET, DETOJET, CITRAJET, TERGAJET, SOLUJET, KEYLAJET

Typical Use: High-volume cleaning using washer-sanitizers, ware-washers, conveyor-washers, or spray and pressure washers.

Advantages: Fast, effective, high volume cleaning.

Concerns: Capital cost; article's ability to withstand machine washing conditions.

Directions: Load articles into racks so that open ends face toward spray nozzles. Place difficult-to-clean articles with narrow necks and openings near the center of the rack, open side down, preferably on special racks with spray nozzles pointing directly into them. Minimize touching between articles. Group small articles in baskets to prevent dislodging by spray action. Use only low-foaming detergent per machine manufacturer dose instructions. If no instructions, use a 1% solution or 1 oz. per gallon of wash water. Use more or less as needed. Use hot

water (above 140°F or 60°C). Most machines have at least three rinse cycles (see Rinsing). Refer to machine manufacturer's directions.

7. Automatic Syphon Pipette Washing

Recommended Products: ALCOTABS

Typical Use: Washing pipettes in laboratories.

Advantages: Effective batch pipette cleaning.

Concerns: Pre-soak pipettes for best results.

Directions: Completely immerse pipettes immediately after use in a pre-soak solution. When ready to clean: Drop an ALCOTAB into bottom of washer. Place pipettes in holder into the washer. Turn on cold or warm water at a rate that will fill the washer and completely cover all pipettes, then drain to the bottom during each cycle. Run water until ALCOTAB has completely dissolved; continue running water to rinse thoroughly (may take an hour to complete washing and rinsing). For analytical or tissue culture work, use distilled or deionized water for final rinse.

8. Rinsing

Don't neglect the rinse! Use ambient, warm, or hot water. A running water rinse directly contacting all surfaces for at least 10 seconds on each surface is desirable. If not practical, use a series of three or more agitated soak tanks or at least two counter-flow cascade tanks to rinse instead.

Specific Cases:

- **Large surfaces** — Several passes with a clean cloth or sponge soaked with rinse water followed by a clean, dry, absorbent wipe can work.
- **Machine cleaning** — After washing there should be at least three rinse cycles. Tap water is suitable for many rinsing applications.
- **Medical and surgical instruments** — Give a final rinse in distilled or deionized water.
- **Laboratories** — Rinse tissue culture and analytical ware with deionized or distilled water. Rinse trace organic analytical ware in distilled or organic-free water. Give trace metal or inorganic analytical ware a final rinse with deionized water.



For the highest levels of critical cleaning only freshly made up solutions should be used for cleaning to avoid any potential for cross contamination.

- **Pharmaceutical equipment** — Rinse according to good manufacturing practice with whichever is required: potable, deionized, distilled, sterile, pyrogen-free or injectable water.
- **Electronic circuit boards and non-conducting electronic devices** — Rinse with deionized water.
- **Sensitive optical or precision manufactured parts** — May require final rinses in deionized or distilled water.
- **Food processing equipment** — Rinse with potable water.

9. Drying

Drying can effect residues and corrosion. Impurities from rinse water can be deposited during evaporation. To minimize this, dry with techniques that physically remove rinse water from the substrate: absorbent wiping, forced air or air knives, azeotropic solvent drying such as isopropyl alcohol final rinse and dry, or vacuum drying, which may also evaporate residues. Water — particularly high purity rinse water — can be corrosive to metal substrates during heated and air-drying. The use of physical removal drying techniques or the addition of corrosion inhibitors (with the tolerance of corrosion inhibitor residues) to the rinse water can help minimize corrosion.

10. Bath Life Extension and Control

For the highest levels of critical cleaning, especially to avoid cross contamination, only freshly prepared solutions should be used. For industrial cleaning applications, however, bath life can be extended while still achieving high levels of cleaning.

Bath life extension techniques:

- Filtering particulates
- Cooling and settling of sludge
- Cooling and skimming oils
- Adding half again as much detergent as the initial load after partially depleting the cleaning life of a bath

Conductivity, pH and % solids by refractometer can be used to measure bath detergent concentration. In general, a pH change of 1 unit toward neutral

indicates an exhausted cleaning solution. Under frequent daily use, detergent solutions can rarely be used more than a week, even when being extended.

Free-alkalinity titration can be used to extend bath life where the soils deplete free alkalinity, as follows:

- Titrate a fresh solution to determine free alkalinity
- Titrate the used solution to determine the percent drop in free alkalinity
- Add detergent to the used bath to bring the free alkalinity back to the new-solution level

For example if your initial solution contains 100 ml of cleaner concentrate and there is a 25% drop in free alkalinity, try adding 25 ml of cleaner concentrate to recharge the solution. Perform a new free-alkalinity titration the first few times to confirm that the detergent is linear with respect to free-alkalinity depletion. This bath-life extension cannot be repeated indefinitely: sludge will eventually form, requiring a fresh solution.

11. Corrosion Inhibition

Corrosion during cleaning is accelerated by the same things that accelerate cleaning: heat, aggressive chemicals, time, and agitation. To reduce metal corrosion, in approximate order of effect, use less heat, corrosion inhibited detergents, lower pH or pH appropriate detergents, shorter cleaning time, and less agitation. Avoid mixed metals in the same bath that form batteries and deposit galvanic oxides. The following techniques may work to limit corrosion, based on the material and/or process involved:

- **Metal:** Use the mildest pH detergent and avoid mixed metals in the same bath.
- **Aluminum:** After abrasion exposes pure metal surface, allow air passivation time prior to cleaning. Use mild acid cleaners such as CITRANOX or CITRAJET to avoid alkaline attack.
- **Plastic:** Use less aggressive cleaners, containing less solvent or surfactant character. Or use lower concentrations of those cleaners, lower cleaning temperatures, less contact time, and less agitation. For stressed polycarbonate and acrylic use surfactant-free DETOJET for cleaning. Unstressed material is not a concern. Avoid alkaline cleaners on polyurethane.



Depending upon the method selected, cleanliness can be detected within three ranges: Greater than or equal to 0.01 grams per square centimeter (g/cm^2), between 0.01 and .001 g/cm^2 , and below 1 microgram per square centimeter.

- **Mild sensitive steel:** Avoid “flash rusting” by rinsing with cold water and using rapid water-removing drying techniques such as dipping in isopropyl alcohol to form an evaporating azeotrope that removes water safely, centrifuge drying, wipe drying, air knives, and drying with oxygen-free gas such as dry nitrogen. Or add a suitable corrosion inhibitor to the rinse water, as long as you can tolerate corrosion-inhibitor residues. Do not use evaporative drying such as air drying or oven drying.
- **Sensitive steel:** Clean with an inhibited cleaner and isopropyl alcohol rinse (or add a corrosion inhibitor to the rinse water).
- **Galvanic corrosion:** Avoid mixed metals in the same bath that can form a battery and deposit oxides on one of the metals. For example, many metals will plate out their oxides on aluminum if the two metals are cleaned in the same bath. Intact stainless steel is generally OK as a mixed metal, but iron, steel, brass, aluminum, bronze and other metals can be a problem mixed with other metals.

12. Cleaning Measurement Procedures

Depending upon the method selected, cleanliness can be detected to varying degrees within three ranges: Greater than or equal to 0.01 grams per square centimeter ($\geq 0.01 \text{ g}/\text{cm}^2$), 0.01–.001 g/cm^2 , and — the highest level of cleanliness measurement — below 1 microgram per square centimeter ($<1 \mu\text{g}/\text{cm}^2$). The sensitivity of each method appears in parentheses after its name.

Water-break (10s of mg/cm^2 level)

Use running water and let it sheet across the surface. Observe if any breaks in the water occur due to hydrophobic (water-fearing) residues. (See “The Hydrophobic Surface Film by the Water Break Test,” ASTM Method F 22–65, at www.astm.org.) The water break test is a fairly crude method, suitable for detecting films of process oils and heavy fingerprints. It will not readily detect non-hydrophobic residues. This test is often used for parts washing; it may not always be suitable for precision cleaning applications.

Atomizer (10s of mg/cm^2 level)

A variation of the water-break test, this requires

observing whether a gently sprayed water mist deposits uniformly or whether water repulsion occurs (usually due to a hydrophobic soil). The atomizer test is slightly more sensitive to hydrophobic soils than the water break test. In the water break test the kinetic energy of the flowing water may overcome a hydrophobic residue, whereas in the atomizer test you may be able to see the results of a little droplet of water being repelled by a hydrophobic contaminant.

Oil-soluble fluorescence (mg/cm^2 level)

In this test the cleaned part is dipped into a fluorescent oil. If oily residues are present, they will be extracted into the fluorescent oil that can be easily observed under ultraviolet light. Of course, using oil-soluble fluorescence requires cleaning the part again after each test. Oil-soluble fluorescence is a highly sensitive method and can give very dramatic visible results of oil residue. The problem with this test is that it really only shows how well your process removed oily soils; it does not reveal particulate or inorganic soils.

Non-volatile residue (10s of mg/cm^2 level)

Extract contaminants from a part using a volatile solvent and evaporate the solvent in a pre-weighed container. Weigh again after evaporation to detect the weight of volatile residues.

Extraction (mg/cm^2 level)

An excellent method for detecting detergent residues. Use a solvent-soaked glass filter paper or polyester swab to wipe surface. Extract or digest filter paper. Use trace analysis on the extract. Can be quantitative if you wipe a known area. Extraction method can be highly sensitive to a wide range of possible soils and residues. The limitations of this method are that an appropriate solvent has to be used to extract whatever type of soil might be there. For example, trace analysis for detergent residues would require water as the solvent. The sensitivity of an extraction test depends on your method of trace analysis. The types of trace analysis often used are UV visible spectrophotometry, total organic carbon (TOC) analysis, high performance liquid chromatography (HPLC), atomic absorption (AA) or inorganic residues, and liquid chromatography (LC) and filtration with microscopic filter examination.

Oil evaporation (10s of mg/cm^2)

For filmy residues, a few drops of organic solvent



Since the properties of various contaminated surfaces are reflected by different contact angles, contact angle testing can be used in a range of processes to determine cleanliness.

can be deposited on the surface and then removed via pipette and placed on a watch glass. If any filmy residues are present, you should observe a characteristic ring of organic-material deposits.

Gravimetric (mg/cm² level)

With small parts of known weight, the amount of excess weight indicates the amount of soil present.

Surface energy (mg/cm² level)

Any hard, flat material has a characteristic surface energy. As a result, depositing a known volume of pure liquid (typically deionized water) will form a droplet of predictable size based on the amount of that energy, and measuring that droplet size will determine surface cleanliness. Generally, hydrophobic soils create smaller droplets; hydrophilic soils, larger ones. The surface-energy test is far more sensitive than the atomizer or water-break test and has the advantage of being able to detect both hydrophilic and hydrophobic soils. The problem with the surface energy test is it only tests the surface directly underneath the droplet where you are measuring. If you have a broad, large surface where measuring one small area would be representative of the cleaning done over the entire surface, this can be an excellent and highly sensitive method. If you have a part with cracks, crevices and holes, the accessible surfaces may not be representative of soils hidden in the various cavities.

Contact angle

A variation on surface energy testing, contact angle is defined simply as the relationship of certain forces when a liquid stabilizes on a part's surface. Since the properties of various contaminated surfaces are reflected by different contact angles, contact angle testing can be used in a range of processes to determine contamination levels, predict cleanliness and adhesive bond strengths, and monitor cleaning operations.

Whether you are checking the moisture effects on silicon wafers or LCD quartz panel glass/metal adhesion, all that is needed is an understanding of the basic theory involved and proper measurement techniques. Contaminants that can be detected by changes in the contact angle include vapors from the process itself (e.g., vacuum or diffusion pump oils), various process chemicals, and even human perspiration. Contact-angle measurement is

especially suited to manufacturing operations that require a nondestructive cleanliness test.

For example, LCD panel surfaces contaminated with organic matter will be less accepting of films such as metals and protective layers, resulting in poor manufacturing yields. Contaminant sources include the vapor of process materials, chemicals, and human perspiration.

Very thin organic contaminants several monolayers in thickness (greater than 10 angstroms) can be evaluated using the contact angle technique. In fact, it is generally agreed that the wetting behavior involves only the last layer or two atoms on either side of a solid's interface. The water contact angle correlates the cleanliness of the surface to the adhesion of the copper deposited onto the surface of the LCD.

Accepted methods of measuring contact angles to determine surface characteristics include the inverted bubble, Wilhelmy Plate, and sessile drop techniques. The latter is the most widely practiced quality control technique, as it is relatively quick and requires minimal investment in time and financial resources.

Follow these tips from *Precision Cleaning* magazine, (Oct. 97, p. 23) to increase the repeatability and validity of contact angle measurement results:

- Use gloves when handling the measurement samples. Organics such as finger oils, cosmetics, and other contaminants will skew contact angle results.
- Note the nature of the droplet after applying it to the surface. Wait until the droplet ceases its advancement and change in lateral movement stops. Measure this time interval and make sure to wait the same period of time after every measurement. Retain consistent time intervals between the placement of the droplet and its measurement.
- Use a consistent measuring liquid — ideally, medical-grade, ultra-purified deionized water from a laboratory supply house. This will limit the number of measurement variables.
- Use test liquids of larger surface tension than the solid's surface energy in order to obtain easy-to-read results.



Whether you are checking the moisture effects on silicon wafers or LCD quartz panel glass/metal adhesion, all that is needed is an understanding of the basic theory involved and proper measurement techniques.

- Neutralize the effects of static charges on substrates. Substrates that are electrostatically charged can skew contact angle readings up to 5 degrees.
- Accurately control liquid droplets so that they are repeatedly deposited onto the sample. Gently move the sample to the liquid droplet formed at the end of the syringe/dispenser in order to minimize gravitational effects.
- In the case of very high contact angles, you may find it difficult to adhere the droplet from the needle to the solid sample. Use a PTFE-coated needle of a higher gauge (smaller inner diameter).
- In case of very low contact angles, use the highest possible needle gauge (smallest inner

diameter needle) to deposit very small droplets onto the sample.

- Other methods such as visual examination using a stereomicroscope, fiberoptic light, or black light are also useful in determining the existence of cleaning process residues.

For extreme low level residue detection at the many $\mu\text{g}/\text{cm}^2$ level, surface analysis techniques such as Auger electron spectroscopy (AES), electron spectroscopy for chemical analysis (ESCA), fourier transform infrared (FTIR), secondary ion mass spectroscopy (SIMS) and optically stimulated electron emission (OSEE) are used.





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