
CLEANING & MONITORING PROCEDURES





Optimizing Your Critical Cleaning Processes Begins with Using the Right Procedures—And Proper Monitoring of Cleaner Performance to Ensure Desired Results

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

In any industrial or processing cleaning “clean” means clean enough to avoid later problems. The key question is: What will happen if the surface isn’t clean? A closely related question, of course, is how can I ensure that cleaning standards are maintained? Whether or not cleaning validation is mandated in your manufacturing operation, it is useful to understand the testing methodologies commonly used for determining cleanliness of industrial parts of the equipment used to process product.

1. Mixing Directions for Alconox Detergents

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

2. Soaking

Recommended Products: ALCONOX, LIQUI-NOX, CITRANOX, TERG-A-ZYME, ALCOJET, DET-O-JET, LUMINOX, DETERGENT 8, and CITRAJET

Typical Use: To clean small items—hospital catheters and tubes, small metal parts and large tank interiors, including pharmaceutical and other blending tanks. An excellent pre-treatment method for loosening soils and preventing 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, LIQUI-NOX, CITRANOX, TERG-A-ZYME, ALCOJET, DET-O-JET, LUMINOX, DETERGENT 8, and CITRAJET

Typical Use: For 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 difficult-to-reach areas requiring pre-soak, ultrasonic, or machine cleaning.

Directions: Make up cleaning solution as in 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, LIQUI-NOX, CITRANOX, TERG-A-ZYME, ALCOJET, DET-O-JET, LUMINOX, DETERGENT 8, and CITRAJET

Typical Use: To clean 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 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).

Product	Form	Dilution (%)	Recommended Amount:	Min Wash Temp	Usual Wash Tem	Protective Gloves	Eye Protection
			(a)oz/gal, (b)gram/l, (c)ml/l				
ALCONOX	powder	1	(a) 1 1/4, (b) 10	Ambient	Warm	Desired	Desired
TERG-A-ZYME	powder	1	(a) 1 1/4, (b) 10	Ambient	Max 130° F	Desired	Desired
LIQUI-NOX	liquid	1	(a) 1 1/4, (c) 10	Ambient	Warm	Desired	Desired
CITRANOX	liquid	1-2	(a) 1-3, (c) 10-20	Ambient	Hot	Required	Required
DETERGENT 8	liquid	2-5	(a) 2-6, (c) 20-50	Ambient	Hot	Required	Desired
LUMINOX	liquid	2-5	(a) 2-6, (c) 20-50	Ambient	Hot	Required	Desired
ALCOJET	powder	1	(a) 1 1/4, (b) 5-10	Warm	Hot	Required	Desired
DET-O-JET	liquid	1	(a) 1, (c) 5-10	Ambient	Hot	Required	Required
ALCOTABS	tablet	-	(1 tablet per use)	Ambient	Ambient	N/A	N/A
CITRAJET	liquid	1-2	(a) 1-3, (c) 10-20	Ambient	Hot	Required	Required

5. Clean-in-Place

Recommended Products: ALCONOX, LIQUI-NOX, CITRANOX, TERG-A-ZYME, ALCOJET, DET-O-JET, LUMINOX, DETERGENT 8, and CITRAJET

Typical Use: For pipe, tank, and filtration systems.

Advantages: Assures clean systems without disassembly.

Concerns: Good circulation in system.

Directions: Make up cleaning solution as in mixing directions. Circulate solution slowly for at least 1/2 hour. Allow several hours for large systems (thousands 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: ALCOJET, DET-O-JET, LUMINOX, DETERGENT 8, and CITRAJET

Typical Use: For 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 towards 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 as 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 below). 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 rinse tanks or at least two counter-flow cascade rinse tanks. For large surfaces, several passes with a clean cloth or sponge soaked with rinse water followed by a clean, dry, absorbent wipe can work. In machine cleaning, after washing there should be at least three rinse cycles. Tap water is suitable for many rinsing applications.

Give medical and surgical instruments a final rinse in distilled or deionized water. In 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. Rinse pharmaceutical equipment according to good manufacturing practice with whichever is required: potable, deionized, distilled, sterile, pyrogen-free or injectable water. Rinse electronic circuit boards and non-conducting electronic devices with deionized water. Sensitive optical or precision manufactured parts may require final rinses in deionized or distilled water. Food processing equipment must be rinsed 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 such as absorbent wiping, forced air or air knives, azeotropic solvent drying such as isopropyl alcohol final rinse and dry, or vacuum drying that may also evaporate residues. Water, and 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 Monitoring, Extension and Control

For the highest levels of critical cleaning only freshly made up solutions should be used for cleaning to avoid any potential for cross contamination. For industrial critical cleaning applications high levels of cleaning can still be achieved with extended bath life. In general, a pH change of 1 pH unit towards neutral indicates an exhausted cleaning solution. Bath life can be extended by physical filtration of particulates and cooling and settling of sludge and skimming of oils. Bath life can be extended by adding one half as much detergent of the initial load after partially depleting the cleaning life of a bath. Under frequent daily use, detergent solutions can rarely be used more than a week even with these bath life extension techniques. Conductivity, pH and, % solids by refractometer can be used to control bath detergent concentration.

Free alkalinity titration can also be used to control bath life of alkaline cleaners where the soil being cleaned depletes free alkalinity—as is often the case with oily soils. Titrate a fresh solution to determine new solution free alkalinity. Titrate your used solution to determine the percent drop in free alkalinity. Add more detergent to the bath to bring the free alkalinity back to the new solution free alkalinity. For example if your initial solution is made up with 100 ml of cleaner concentrate and you observe a 25% drop in free alkalinity, you should try adding 25 ml of cleaner concentrate to recharge your solution. You should perform a new free alkalinity titration to confirm your recharge the first few times you use this recharging method to be sure that the detergent you are using is linear with respect to free alkalinity depletion. This form of bath life extension cannot run indefinitely, sludge will eventually form. Fresh solutions must be periodically made up.

11. Corrosion Inhibition

Corrosion during cleaning is accelerated by the same things that accelerate cleaning: heat, aggressive chemicals, time, and agitation. In approximate order of importance, to reduce metal corrosion concerns, use less heat, lower pH detergents, shorter cleaning time, and less agitation. In general use the mildest pH detergent to limit metal corrosion. Higher pH detergents such as ALCOJET have special corrosion inhibitors that often allow it to be used successfully to clean soft metals such as aluminum.

In approximate order of importance, in general to reduce plastic corrosion, use less aggressive cleaners that have less solvent or surfactant character or use lower concentrations of those cleaners, use lower cleaning temperatures, use less contact time, and finally use less agitation. With aqueous cleaning metal corrosion can occur during rinsing and drying. Corrosion inhibitors can be added to rinse water provided that any associated inhibitor residue does not interfere with the surface being cleaned. Keeping the surfaces cleaned hot with hot rinse water and using rapid heat or vacuum drying can accelerate drying and minimize metal corrosion. Forced air drying and air knives that physically remove rinse water can minimize drying corrosion. Drying with hot oxygen-free gas such as nitrogen can also control corrosion during drying.

With mild steel you can have "flash rusting" when you rinse with hot water and dry with hot air. In some instances, by lowering the water temperature or drying temperature, corrosion can be avoided on mild steel. For instance in a case where flash rusting on mild steel had been occurring using 150°F rinse water and ambient air drying, it was found that flash rusting could be avoided by using 120°F rinse water in place of the 150°F rinse water.

12. Cleaning Measurement Procedures

In any industrial or processing application “clean” means clean enough to avoid later problems: What will happen if the surface isn’t clean? Answering this question, you can then choose the best testing methods for measuring cleanliness and assuring product quality:

- **Water-break**—(10s of mg/cm² 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—found at www.astm.org.) The water break test is a fairly crude test that is 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/cm² 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 where 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**—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 ultra-violet 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/cm² level) extract contaminants from a part using a volatile solvent and then evaporate the solvent in a pre-weighed container and then weigh again after evaporation to detect the weight of volatile residues.
- **Extraction**—(mg/cm² level) A particularly 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. If you were doing trace analysis for detergent residues you would use water as your 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/cm²) For filmy residues, a few drops of organic solvent 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 indicated the amount of soil present.
- **Surface-energy**—(mg/cm² level) Any hard, flat material has a characteristic surface energy. As a result, a deposit of a known volume of pure liquid (typically deionized water) will form a droplet of predictable size based on the amount of that energy. Measuring 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 little area of it would be representative of the level of the cleaning that went on the entire surface, this can be an excellent and highly sensitive method. If you have a part that has lots of cracks and crevices and holes, the surfaces that are accessible for you to place a droplet of liquid to measure surface energy may not be representative of the types of soils that may be hidden in any cracks, crevices, or blind holes.

- **Contact-angle**—A variation of surface energy testing is contact-angle—defined simply as the relationship of certain forces when a liquid stabilizes on a part’s surface. This method can be used to determine cleanliness since the properties of various contaminated surfaces are reflected by different contact angles. Contact-angle measurement is especially suited to manufacturing operations such as wire bonding on PCBs or the application of thin films on quartz glass which require a cleanliness test that is nondestructive. Vapors from the process itself, such as vacuum or diffusion pump oils, various process chemicals, or even human perspiration, are all contaminants whose presence can be detected by changes in the contact angle.

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.

Several “real world” examples will help illustrate what can be learned from the use of the contact angle measuring technique to determine the existence of impurities on a surface.

LCD (Liquid Crystal Display) panel surfaces contaminated with organic matter will be less accepting of a variety of films, such as metals and protective layers, resulting in poor manufacturing yields. Sources of such contaminants 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.

Water contact angles can be used in various 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.

As with other cleanliness verification techniques, special tips for contact angle measurement, if practiced, will increase the repeatability and validity of the measurement results. The following tips from *Precision Cleaning* magazine, (Oct. 97 p. 23) were helpfully compiled: —Use gloves when handling the samples to be measured. Organics,

such as finger oils, cosmetics, and other contaminants, will skew the contact angle results.

—Note the nature of the droplet after applying it to the surface. Wait until the droplet has ceased its advancement and no more change in lateral movement has occurred. Measure this time interval, making sure that you wait this period of time after every measurement. Retain consistent time intervals between the placement of the droplet and its measurement.

—Use medical-grade, ultra-purified deionized water from a laboratory supply house in order to have a consistent measuring liquid. 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.
- 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) for controlling very small droplet volumes 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.

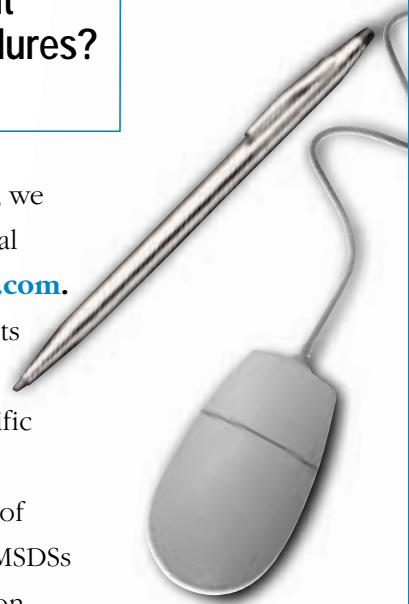
For extreme low level residue detection at the many ug/cm² level, surface analysis techniques such as Auger electron spectroscopy (AES), electron spectroscopy for chemical analysis (ESCA), fourier transform infrared (FTIR), secondary ion mass

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