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Wet Chemistry Electrum Laboratory



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1 General

All handling of chemicals in the Electrum laboratory shall be performed in such a manner, that it at all times protects the operator from all potential hazards, associated with chemicals and experimental processes. Carefully read the chemical's label and Material Safety Data Sheet (**MSDS**) before using a chemical for the first time. All approved chemicals in the Electrum laboratory and their MSDS are listed in LIMS (Info/Chemical list). Know the physical and health hazards associated with the chemicals you are using.

When evaluating potential hazards consider this:

- Hazards may be airborne e.g. gas, fume/vapor or particles i.e. inhalation.
- Hazards may occur in contact with eyes or skin (i.e. splash) or if swallowed (i.e. transferring contamination to food via hands)
- Comply with the Occupational Exposure Limits. The occupational exposure limit (*sv. hygieniskt gränsvärde*) is an upper limit on the acceptable concentration of a hazardous substance in the workplace air, for a particular chemical/material or class of materials. In Sweden this limit is set by "Arbetsmiljöverket" and enforced by legislation to protect occupational safety and health.
- There are dangerous substances for which there exist no formal occupational exposure limits. In these cases, control banding strategies (*sv. försiktighetsprincipen*) can be used to ensure safe handling i.e. they are to be treated as hazardous and the concentration must be zero in the workplace air.
- Comply with the **Flammability Limits.** Also called explosive limits, (*sv. explosiv gräns*) give the proportion of combustible gases in a mixture, between which limits this mixture is flammable. Gas mixtures consisting of combustible, oxidizing, and inert gases are only flammable under certain conditions. The lower flammable limit (LFL) (lower explosive limit) describes the leanest mixture that is still flammable, i.e. the mixture with the smallest fraction of combustible gas, while the upper flammable limit (UFL) (upper explosive limit) gives the richest flammable mixture. Increasing the fraction of inert gases in a mixture raises the LFL and decreases UFL.

You may not introduce previously unknown chemicals without approval from the . Purchase of personnel chemicals should be done through the Electrum Laboratory personnel.

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2 Chemicals

2.1 Standard chemicals and personal chemicals

Electrum laboratory supplies a set of standard chemicals¹ associated with micro and nano-fabrication. All standard chemicals are of VLSI-quality unless only other grades are available.

All non standard chemicals are personal, meaning that they are purchased and used by a user or a specific project. The purchase of these should also be done through the Electrum Laboratory personnel.

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Only chemicals that are approved by the Electrum Laboratory personnel (Laboratory Director) may be used in the cleanroom. If there is a need for a new chemical, that chemical must be presented to the Laboratory Director, together with the supplier's MSDS, Product Data Sheet and a risk assessment of how this chemical will be used in a safe manner.

If the chemical has dangerous properties, and the Lab Director concludes that the risks cannot be lowered by the use of proper tools, procedures and protective gear, processing involving the chemical may be denied. Similarly, Electrum laboratory does not accept the use of dangerous or environmentally unfriendly chemicals, if there are better alternatives.

Fundamental for the safe use of a certain chemical is its properties. That information is attainable from experienced colleagues, laboratory staff, and person responsible for the tool where the chemical is in use and in particular from the Material Safety Data Sheet (**MSDS**) from the supplier.

If medical attention is necessary as a result of exposure to a chemical, the MSDS of that chemical should be presented to the physician.

¹ A Standard chemical is a chemical commonly used by several different user groups/departments or companies within the Electrum laboratory.

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2.2 Risks

In principal, all occurring chemicals in Electrum laboratory are associated with chemical safety risks. Risk, in general terms, relates to an event, the probability of the event occurring and the negative impact of the event. Larger probability of the event occurring and greater negative consequences contributes to the overall risk. One could say that risk is the product of the probability for the event taking place and the negative outcome of the event.

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The above is best illustrated with an example. The wet bench provides a good protection against airborne chemicals, but no protection against splashes. Most wet benches are operated with corrosive chemicals. Splashes can be produced from simple handling mistakes, such as dropping an item into a container. It is fair to assume that a statistically relevant portion of such accidents will produce splashes that reach the face of the operator. This is a serious risk, which is easily reduced by the use of proper face covering protective gear. Splashes may still reach the operator, but the damage it can induce is greatly reduced.

- Processing where it is possible to foresee likely scenarios where a mistake or accident can result in severe consequences can never be tolerated.
- Splashes of a corrosive liquid onto the face can result in permanent injury to the eyesight.
- All incidents and accidents must be reported to the laboratory staff.

Properties and their corresponding symbols of some of the chemicals used in the cleanroom:



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2.3 HF (Hydrofluoric Acid)

HF is highly toxic and corrosive. The toxicity is due to the fluoride ion content of HF. Any solution containing a source of free fluoride ions is also toxic. Chemicals such as HF, NH4F and the mixture of the two (BOE, BHF), may differ in concentration, vapour pressure and volatility, but the fundamental toxicity is the same.

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Fluoride ions readily penetrate the skin, causing destruction of deep tissue layers. Symptoms of exposure to low concentrations can be delayed. Exposure to concentrated HF (50% by weight) cause immediate, severe, burning pain and a whitish discoloration of the skin. The fluoride ions have a high affinity for calcium and magnesium ions. Chemical reaction between them forms water insoluble calcium- and magnesium fluoride salts. Local tissue destruction (at the point of contact) results from free hydrogen ions which cause corrosive chemical burns, and free fluoride ions which cause deep tissue destruction from the reaction with calcium ions in the human cells.

HF acid is essentially a gas dissolved in water. The vapour pressure of HF is high at room temperature, and HF acid will produce toxic and corrosive fumes.

In the body, Ca and Mg ions mediate a variety of physiological processes, as muscle movement and body chemistry. Skin exposure to greater areas (more than 160 cm²) or inhalation of HF vapours, may lead to systemic toxicity. Systemic conditions include hypocalcemia (loss of calcium) and hyperkalemia (too much potassium). Since calcium and potassium regulate the heart rythm, an arrhythmia and cardiac arrest can result. Fatalities have been reported from concentrated HF burns to as little as 2.5% of the total body surface. That is the equivalent of a single hand.

Dilute HF must be treated with the same caution as concentrated HF. Symptoms to exposure can be delayed. Exposure to 5% HF (by weight) may not produce symptoms for up to 24 hours. Treatment will be delayed correspondingly for unnoticed exposure, increasing the risk of local tissue destruction and systemic toxicity.

Calcium gluconate gel (aka "HF antidote") for treatment of HF burns is available in the cleanrooms. Consult the lab specific appendix for locations.

2.4 Acids

Acids are corrosive and will damage human tissue. Especially the eyes are vulnerable to exposure. Acids may only be used in dedicated wet benches and fume hoods. A supply of typical etchants is provided as standard stock in the cleanroom. Acids can induce chemical burns to the skin if exposed, be toxic, cause rapid heating through exothermic reactions (and thermally burn the body) and even initiate explosions.

Examples include: Hydrofluoric acid (HF), Hydrochloric acid (HCl), Sulfuric acid (H₂SO₄), Nitric acid (HNO₃), Ammonium Fluoride NH₄F, Perchloric acid (HClO₄) and Acetic acid (C₂H₄O₂).

2.5 Bases

Bases are corrosive and will damage human tissue. Especially the eyes are vulnerable to exposure, since bases are difficult to rinse off. Bases may only be used in dedicated wet benches and fume hoods. Common bases in the Myfab labs are (Potassium Hydroxide) KOH, (Sodium Hydroxide) NaOH, (Ammonium Hydroxide) NH₄OH, (Tetra methyl ammonium hydroxide) TMAH and photoresist developers (typically containing NaOH or TMAH).

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2.6 Piranha (7-up)

Piranha (or 7-up) is the mixture between sulfuric acid and hydrogen peroxide, usually in a 3:1 ratio. The international nick name for this mixture is Piranha solution.

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Piranha is used mainly to clean wafers from photoresist residues or other organic contaminants. When mixed, sulfuric acid and hydrogen peroxide reacts; the hydrogen peroxide will undergo decomposition, producing highly reactive oxygen radicals that will oxidize most carbon -containing species. The sulfuric acid and hydrogen peroxide are themselves oxidizing agents and the sulfuric acid will act as an excellent solvent for oxidized carbon species. The reaction between sulfuric acid and hydrogen peroxide is highly exothermic, and the temperature of the mixture will initially rise to at least 120 °C.

The Piranha solution combines chemical corrosive properties with high temperature, and is used in dedicated wet bench containers which provide none or little splash protection. The Piranha mixture must be prepared and used with caution, always using proper protective gear.

If hot Piranha reaches the face in an accident, the result will probably be instant, permanent damage to the eyesight and skin burns that will leave disfiguring scars.

2.7 Organic solvents

Isopropanol (isopropyl alcohol – IPA) and Acetone are the most commonly used organic solvents in any microelectronic cleanroom. Isopropanol is a general cleaning agent for both wafers and equipment. Acetone is used to remove resist, clean wafers and resist contaminated equipment, etc. The properties of these solvents, and the effect they may have on humans, are well known. Health related risks are fairly low when handling Isopropanol and Acetone.

However, they are both flammable liquids.

Other organic solvents are used within Electrum Laboratory. Some are more or less toxic, cancerogenic or mutagenic. For some solvents the health affecting properties are not known or fully understood, which may also be true for other chemicals than organic solvents. Such chemicals should be treated as toxic. Always consult the MSDS before starting to use a new chemical. With very few exceptions (e.g. IPA), the use of organic solvents must be confined to ventilated workstations. Not only Isopropanol and Acetone are flammable. For the sake of simplicity, all organic solvents should be considered flammable.

Some organic solvents are known to be cancerogens or toxins. They should be avoided if there are less toxic or cancerogenic alternatives to use. For example, if ethanol could be used rather than toxic methanol in a process, then it should. The cancerogen Benzene can sometimes be substituted with Toluene.

With the use of Isopropanol and Acetone follows a fire risk.

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2.8 Hydrogen peroxide

The only significant standard chemical that cannot be put in any of the above categories is hydrogen peroxide. This chemical is a strong oxidizer and may react violently when mixed with other chemicals. Using hydrogen peroxide in mixtures is therefore limited to well known recipes like 7-up (Piranha solution) or other standard mixtures.

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2.9 Photoresist and other organic chemicals

Special organic chemicals like photoresists, epoxy resins, adhesives, and so on, are also present in the cleanroom. They most often do not have the same acute corrosive or toxic properties as acids. However, if they are handled in a faulty manner over time, this may lead to a long-term exposure to the skin or respiratory system with known or unknown consequences as a result.

Most photoresists are mixtures of solvents, novolac resins and photo-active compounds. The resulting mixture can be toxic, flammable and/or irritating. Use these chemicals only in approved ventilated areas, such as solvent hoods or photoresist spinners.

Fumes evolved during use of photoresists, polymers, etc., must not be inhaled. Dedicated workstations like spinners or bake ovens are fitted with exhaust ventilation; if it smells there is a technical problem with the equipment or the handling procedure.

Epoxy resins are known to induce sensibilisation from prolonged skin exposure. Sensibilisation is an allergy-like condition with low tolerance to expoxy, and possibly other chemicals and allergens.

3 Cryogens

Liquid nitrogen (LN2), Liquid Argon, Liquid Helium and Solid CO2 (dry ice) are examples of cryogens. Cryogenic chemicals present a safety hazard due to their extreme cold. Users should be familiar with this hazard and use appropriate cryogen gloves as well as designated personal protective equipment against the freezing effects. Under no circumstances should a user allow to contact LN2 with their body. Severe injury can result from such contact.

All cryogens listed above can displace the oxygen in the air as they evaporate. Therefore you must only use nitrogen, liquid nitrogen, helium, liquid helium and carbon dioxide in well-ventilated rooms and after having performed an analysis of the amount of air that could be displaced by the cryogen proposed for use. Provided only a small fraction of the air will be displaced, the cryogen can be used safely. Keep the room especially well ventilated during use.

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4 Harmful metals

Some of the metals used in evaporation processes in the cleanroom are harmful and should be treated with high precaution.

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Nickel (Ni) shows limited evidence of carcinogenic effects and may cause sensitization by skin contact.

Chrome (Cr) is very toxic in contact with skin and if swallowed. It is also harmful to inhale. Read the MSDS prior to use!

Working rules:

- Always use protective gloves and visor/goggles (Cr); avoid direct contact to the skin.
- When working with the metal or mechanical cleaning of tool parts (risk for particle generation) take precautionary measures against dispersal and accumulation of metal particles. Work in a fume hood or use respirator (andningsskydd).
- Metal remains and/or contaminated tool parts to be replaced (boats, shields, etc.) should be treated as hazardous waste: wrapped in plastic bag and left to the destruction.
- **Chrome**: in case of contact with eyes, rinse immediately with plenty of water (for 15 minutes) and seek medical advice.
- **Chrome**: in case of accident, if you feel unwell or experience other effects, seek medical advice immediately.

5 Electrical hazard

The cleanroom is fitted with a vast collection of electrical tools. The power consumption in the cleanroom per square meter is significant. The result of an accident involving electric chock may range from discomfort to instant death. Current strength and current path through the body decides the outcome of an electrical chock. An electric current that passes from hand to hand or hand to foot, is most likely fatal if it is in the range 50-500 mA, due to effects on the nervous and muscular system (heart failure). Higher currents normally do not kill instantly, but will burn interior and exterior parts of the body, giving permanent or eventually fatal injuries (failure of internal organs, burns that are complicated by infections, etc.). Interior burns from currents in the range 50-500 mA may also give long term problems locally or to the kidneys, if the hemoglobin coagulates in blood vessels from the heat induced by the current.

Power: It is not allowed for users to perform trouble shooting or repairs on tools.

6 Fire hazard

The combination of chemical usage, flammable process gases, high area density of electrical tools and tools working at elevated temperatures, increases the probability of a fire incident. The outcome of a fire may be severe consequences to the cleanroom and its users as well as the environment surrounding the cleanroom, due to the presence of toxic gases and chemicals.

Fire is a very serious condition, and fire prevention must always be a priority.

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7 Personal protection

- Put chemical protective gloves (yellow or green) over the nitrile/vinyl gloves before you start working with chemicals at the wet benches.
- Use a face protective visor.
- Use an plastic apron.
- Inspect your gloves carefully. If the gloves are discolored or damaged, they should be disposed of and replaced immediately. Rinse the gloves carefully before placing them into the trash bins. New gloves can be found in the storage cabinet behind the Wet chemistry area.
- After the work in the wet bench has been completed, the chemical protection gloves should be rinsed and put back to their designated place, without touching any surface besides the wet bench.
- The protective gloves are used to protect your hands. Assume that they will be contaminated with corrosive chemicals when moving cassettes or holders over the baths. Such contamination should not be transferred to the maneuvering panel of wet benches. Rinse of the gloves with a DIW-gun, remove them and operate the panel with your cleanroom gloves.



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8 Handling of chemicals

The following routines apply to all handling of chemicals in the cleanroom:

- Chemicals may only be handled in a fume hood.
- Apply the Acid-Into-Water rule. AAA- Always Add Acid (sv. SIV- Syra I Vatten)
- Never mix chemicals without prior knowledge of the consequences.
- Some containers/beakers are dedicated for certain chemicals, and may not be used for anything else.
- Hydrofluoric acid (HF) and Potassium Hydroxide (KOH) solutions may only be used in plastic containers.

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- Open containers containing Ammonia (NH₃) and Hydrochloric acid (HCI) may not be placed next to each other, as the chemical reaction between the vapours will generate solid particles in the cleanroom.
- Some chemicals are being re-used. Pour them back into the bottle and carefully rinse the container with pure water.
- Pay special attention while you are working with Hydrofluoric acid (HF). This acid may cause severe burns into the bone marrow, and the symptoms may sometimes not be noticed until 24 hours after the exposure. Any exposed skin should be covered with "HF Antidote Gel". Spread the gel and contact a physician as soon as possible. The paste is available near the process benches that contain HF. Please note that HF is a component of BOE (Buffered Oxide Etch).
- Ensure that flammable chemicals (e.g. acetone or propanol) are not used near hot surfaces. Even small amounts may cause fire incidents.
- You may not introduce previously unknown chemicals without prior approval from the Electrum Lab Director. Purchase of chemicals should be done through the Electrum Laboratory personnel..
- Do not dispose of used chemicals if you are not sure how to do this. Store in closed bottles/containers until further notice. This applies especially to concentrated, unmixed chemicals. Contact the Electrum Laboratory personnel for advice.
- Avoid direct contact with any chemical.
- Never smell, intentionally inhale or taste a chemical.

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9 Conduct

Wet chemistry area is used by many people, often for shorter time by using beakers or different temporary experimental sets. In order to maintain a safe and effective working environment, everybody must have the discipline to clean and sort after themselves. Having a 5S-mindset in this room is of special importance.

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Everybody is obliged to maintain order in this room, by following these guidelines:

- Use protective clothing when handling chemicals.
- Don't leave dirty dish in the sink.
- "Book" the fume hood you want to use, by filling in the information on the "In Use/Free"- card
- Write the date on the designated card when you fill a fresh chemical into a wet bench bath
- Clean the workplace (fume hoods, wet bench or desk) directly after finished work.
- Return cassettes, handles etc to their dedicated location.
- Don put/leave anything on top of the wet benches.
- Treat waste disposals as described in the instruction acr050781-Waste fractions in the cleanroom.
- Containers, used beakers, experimental set etc with chemicals shall be clearly labeled with name, telephone number, chemical contents and date.
- Chemicals in containers that are not clearly labeled should be disposed of immediately. Mark the container "unknown content" and contact the Electrum Laboratory personnel.
- After the work in a wet bench has been completed, the chemical protection gloves should be rinsed and put back to their designated place, without touching any surface besides the process bench.
- The user is responsible for cleanup of minor chemical spills. Please contact the Electrum Laboratory group if a major spill has occurred.
- Do not disturb persons working with chemicals.
- Respect you colleagues by following these guidelines!

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10 Chemical cupboards

Chemicals and chemical wastes have to be store in ventilated cupboards. Only compatible chemicals are allowed to be stored in the same cupboard. There are mainly two different types of cupboard that are assigned for storage of chemicals of three different groups:

- a) Acid cupboards: Chemicals that are inorganic acids, base, oxidizing or water based chemicals, as well as non-toxic inorganic salts should be stored in such cupboards. These cupboards are connected to the acid exhaust system.
- b) Solvent cupboards. These cupboards are assigned for storage of only organic solvents. Solvent cupboard are connected to the solvent exhaust system

If you want to store a mixture of different chemicals contact the Electrum Laboratory personnel for further assistance. There are dedicated cupboards for bases and photoresists in the lithography rooms.

11 Ventilated work places

Wet benches and fume hoods represent two different technical solutions to the problem of handling chemicals in a safe manner.

Generally speaking, a solution to this problem should contain:

- A way to separate the user from any harmful airborne pollutants.
- A surface to work on.
- A way of disposing of the chemicals after usage.
- A choice of materials that is compatible with the intended chemicals.

11.1 Fume hoods

Fume hoods are designed to protect people from fumes and chemical splashes. The fume hood is an exhaust ventilated work space with a see-through height adjustable sash. The exhaust ventilation will constantly draw air into the fume hood. If the air velocity in the hood opening is at least 0.5 m/s, airborne chemicals will not escape into the room. The sash gives an excellent protection against splashes. Typically fume hoods require airflows in the range 500-800 m³/h, depending on hood geometry and design principle.

A traditional fume hood operates with fixed airflow. This means there is a damper on the exhaust ventilation duct behind the fume hood. The damper is adjusted to a fixed position, so that the air velocity is at least 0.5 m/s when the sash is raised to a certain standard height. If the sash is raised further, the air velocity will drop. If the sash is lowered, the velocity will increase.

Regardless of design, all fume hoods must be fitted with ventilation guards that monitor the air velocity (directly, or indirectly by measuring the pressure in the hood). If the velocity decreases to an unsafe level, an audio alarm signal is normally given.

Fume hoods are connected to one of two available exhaust systems, acid exhausts in PVC and solvent exhausts in sheet metal. (Check local conditions). When working with large volumes of chemicals, fuming chemicals or at high temperatures it is necessary to use a hood with the correct extract system. For small volumes at room temperature it is not essential to use the fume hood.

There are typically two independent drainage systems in fume hoods:

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• The acid/base drainage is used for acids and bases, and DI water. The waste is drained to a neutralizing tank on the floor below the cleanroom.

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• The solvent drainage is used for organic solvents (possibly with exception for halogenated solvents, consult with Electrum Laboratory personnel). The waste is drained to a waste storage tank.

Keep in mind the following fume hood working principles:

- Respect the ventilation guard. If the alarm is on, the fume hood provides an insufficient protection against airborne chemicals. Low exhaust capacity in the fume hood may be locally induced (sash is opened to high) or due to a problem with overall capacity (failed exhaust fan).
- Use the fume hood with the sash opened to the minimum height your work requires.
- Always use the sash as splash protection. That is, position the sash so that you work looking through it.
- Do not place beakers or containers with chemicals closer than 15 cm to the front edge of the fume hood. This simple rule together with proper use of the sash ensures good protection.
- The cross sectional geometry of large containers or equipments placed in the fume hood can result in insufficient airflow in front of the object. Air will escape above and on the sides of the object, but fumes may diffuse out from the objects front. If the object is placed on 5 cm spacers, air will flow also beneath the object, improving the protection.
- **Mind the fire hazard!** Flammable chemicals must be handled with caution. Consult the MSDS for properties of the chemicals you are handling. Organic solvents are always flammable. Do not leave beakers or containers with organic solvents unattended together with possible sources of ignition like electrical equipment, especially not hot plates. A fire in a fume hood will initially not be detected by the smoke detectors, since the smoke effectively will be removed by the exhaust. If the fume hood is connected to a PVC duct, the fire can propagate through the exhaust ducts.
- Mind the safety of other users! Clean the work area after using the fume hood. Never leave chemicals, beakers, warm hot plates etc unattended in the fume hoods. If it is necessary to leave before the process is complete, a note indicating name, telephone number, chemical contents and date must be clearly visible.

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11.2 Wet benches

The wet bench is an exhaust ventilated work table. The height and depth of the wet bench are suitable for working in an upright position. The top surface is perforated and fitted with recessed chemical baths suitable for cassette handling of wafers. Air flows through the perforation. The baths are covered by lids that open in a flip-up fashion. When a lid is open, air flows through an open section surrounding the container. Some benches have a ceiling attached screen above them, and HEPA-filters are installed in the ceiling area onto which the bench is projected. The screen and the filters will provide clean air to the bench surface, and if the air supply is in balance with the bench exhaust, vapours are effectively removed. The wet bench provides a much cleaner handling of wafers than the fume hood. The latter draws air from the cleanroom ambient in an uncontrolled manner; the air is not drawn directly from the HEPA-filters, and will pass the operator, picking up particles.

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Wet benches provide good protection against harmful vapours from acids (or other liquids) in the containers, but are less effective for chemicals handled on top of the work surface. The maximum safe working height is 200 mm. Above the stipulated maximum working height, vapours from the container can escape into the room. A safe working distance from the bench edge is 15 cm or more.

Work is performed with chemicals in the line of sight, without any see-through screen in between. If an item is dropped into a container, splashes will be the result. Since all wafers are handled by cassettes or wafer holders, there is certainly a risk potential in the bench construction and operation. An apron should also be used.

If the perforation in the top surface is covered by papers, beakers and other items, the airflow through the bench will be reduced. The air supplied to the bench from the filter ceiling is constant, and will in this situation exceed the exhaust flow. The excess air will "roll" over the front edge of the bench, possibly bringing with it vapours from the bench chemicals.

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Common functions in wet benches are drain valves, heated chemical baths, ultrasonic baths, etc. These functions are operated from a maneuvering panel on the bench front. The protective gloves are used to protect your hands. Assume that they will be contaminated with corrosive chemicals when moving cassettes or holders over the baths. Such contamination should not be transferred to the maneuvering panel. Rinse of the gloves with a DIW-gun, remove them and operate the panel with your cleanroom gloves.

- Observe that the wet bench provides no protection against splashes at all.
- Face covering shield and protective gloves must be worn when operating a wet bench.
- Cleanroom gloves must not be considered as chemically protective gloves.
- The top surface should be kept free from unnecessary items.
- Do not touch the manouvering panel with protective gloves on.

12 Drain

There are two drain systems installed in the lab, solvent drains and acid/base drains. The solvent waste is collected via stainless piping's into a stainless steel tank. Waste water in the acid/base drain is passed through a pH neutralization facility, before being expelled to the public sewer.

All process benches and all fume hoods are connected to the proper waste drain. If you have waste chemicals and you are unsure about what to do with them, please contact the lab personnel for advice. Never discharge any materials in the drains if you are not sure it belongs there. Put it in a closed bottle so you can decide later. Some chemicals should always be collected in waste bottles after use, to be disposed of by the lab personnel.

- The acid/base drain tubing is fabricated in plastic, why draining of hot baths should be avoided.
- In fume hoods with both acid/base and solvent drain there is a risk of draining acid into the solvent drain, by mistake. This will corrode the welding in the solvent tank, eventually leading to costly repairs. A greater volume oxidizing acid (sulfuric or nitric acid) may also start a violent reaction in the solvent tank.
- The chemical wastes which are left for further destruction should be labelled and identified by full name of the chemical(s), concentrations, hazard symbol and name of user.

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13 Exhaust

The cleanroom have two or more separate exhaust ventilation systems. One system for corrosive applications made of plastic. The other system is made of sheet metal, for applications that are non-corrosive and work at high temperatures, uses flammable gases or organic solvents.

Failure of an exhaust fan will turn the cleanroom into an unsafe work environment. Any sign of low capacity or failure on the exhaust must be reported to lab staff. Tools that require exhaust and are fitted with alarms or interlocks, must not be tampered with and operated despite any indication of exhaust failure.

The exhaust fans are powered by a back-up diesel powered generator in case of a power failure. The main exhaust flow is through the fume hoods, wet benches and tools using toxic or flammable gases often at high temperature (Metalorganic vapour phase epitaxy (MOVPE), Low pressure chemical vapor deposition (LPCVD), etc).

This cleanroom have a static fixed position on the dampers and the exhaust capacity has an upper limit. Fume hoods, lids to chemical baths in wet benches, doors to exhaust ventilated cabinets, etc, should not be left wide open.

14 General working guidelines

14.1 Where are the chemicals?

All chemicals in the wet chemistry room are stored in ventilated chemical cupboards. One cupboard is dedicated for acids and the other for solvents, see <u>chemical cupboards</u>. If you cannot find the chemical you intend to use in the storage cupboard(s) check the pass-through cupboard.





Picture 1

Picture 2

Before handling any chemicals use required personal protection equipment, see Personal protection.

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Picture 3

7 W21 KOH 6" Quartz bath with he

6" Water bath with heating

Picture 4

The pass-through cupboard is the point of delivery for chemicals to the wet chemistry room and is not intended as storage.

Remove the plastic bag wrapped around the bottles and transfer all the chemicals from the pass-through cupboard to the proper storage cupboard.

If the (standard) chemical you intend to use is not available in the cleanroom, you can order it by writing the name of the chemical and quantity on the whiteboard, located on the entrance to the service room.

14.2 Which wet bench to use?

All wet benches and fume hoods are tools registered in LIMS with a unique name, sorted under the category "wet process benches". They are optionally bookable, and you can find information regarding their technical configuration and user instructions in LIMS.

There are six wet bench clusters in the wet chemistry room, dedicated for etching different materials. "MEMS" is for etching of Si, "Oxide" for etching of SiO₂, "Nitride" for etching of Si₃N₄, "7-Up" for removal of photoresist on Si/SiO₂, "Metal" for etching of metals (like AI and Au), "Strip" for lift-off and non lift-off processes using solvents.



ofSi

Observe that every wet bench cluster have dedicated processes and therefore dedicated cassettes, marked with a color marker (picture 6). This color code is indicated on the sign, located above the wet bench, which describes the properties of every bath in that wet bench.

In the example of the "MEMS"- bench, the color marking on the cassettes to be used is brown, as is the circle in the upper left corner on the sign (picture 5).

Some baths are made of quartz others of PVDF or stainless steel. Some baths are for wafers up to 6" others 4". Some have heating, stirring or ultrasonic, others don't. This information is also shown on the signs of every wet bench cluster and in LIMS.

Picture 6

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Picture 7

Identify the bench and bath suitable for your process. If the bath is already full, check the date when the chemical was poured (picture 7). If the chemical was changed recently, there is no need to exchange it. If you want to empty the bath and pour fresh chemical(s) into the bath, use the aspirator to suck out the old chemical, in case the bath doesn't have a drain. Rinse the aspirator with DI- water after you are done.



Caps on some bottles are difficult to open, but there is a "cap twister" that makes this operation easier (picture 8).

Make sure that no parts of cap falls down in the drain, that can cause a blockage in the pipe.

Picture 8



Picture 9

Use caution when pouring chemicals and try to avoid splash. The rectangular bottles can initially be held as shown in picture 9 to minimize splash. Don't cover the perforated surface of the wet bench as this will reduce the exhaust.

After you have poured fresh chemical(s) into a bath, wipe out the old date (from the sign shown in picture 7) with a cloth damped with isopropanol, and write the date you filled the bath, using a permanent marker.

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The volume differs between baths, but the chemical should cover your substrate entirely.

The important thing to remember is that the level of the chemical in the heated baths must reach the gray edge, which correspond to approx. 8 liters of liquid. Also make sure that the temperature probe is submerged in the chemical. See picture 10

Ignoring this may lead to heater damage or fire.

Picture 10



Picture 12

Picture 11

Chemical containers and bottles must be rinsed with water before being disposed of. Rinse manually (fill the bottle twice with water) or use the bottle rinse (picture 11). If the bottle rinse is busy you must either rinse manually or return later to complete the rinse procedure of your bottles. It is not ok to leave bottles on adjacent benches and expect other users to process them. Always rinse the exterior of the bottle/container with a DI-water pistol

(especially the threads on the neck and the bottom). Rinsed glass bottles are collected in the

glass bin. Rinsed plastic bottles are collected in the container for plastic waste (picture 12). It is important that only clean plastic bottles are placed in the container for plastic waste. The cap must be separated from the bottle when disposed of.

For more details see instruction: "Chemical waste handling" in Quality Manual.

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Picture 14

Picture 13

After you have etched your wafers, rinse them together with the process cassette and the handle, in the bubbler for 10 minutes. Rinse the handle with a DI-water gun or remove the handle from the cassette and place it into the bubbler next to the cassette, so the entire handle gets rinsed.

Transfer the wafers to a dedicated rinse/dryer cassette and run it. Return the cassettes and handles to their designated and marked place.

Don't forget to turn off the heater in the heated baths after you are done!

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14.3 Ultrasonic bath

The ultra sonic bath must only be filled with water. Make sure that the ultrasonic bath is filled with water up to the minimum level. Place your samples in a separate beaker filled with the desired solvent. Place the beaker in the ultrasonic bath. Turn on timer and heater if needed.

14.4 Which fume hood to use



Fume hoods named "FH wet chemistry-1... 6" are equipped with a DI- water gun, solvent drain and a water/acid drain. In order to use them fill in the card shown in picture 15. Read more about fume hoods here <u>Fume hoods</u>.

You control the valves for the water/acid and solvent drains on the control panel individually. In order to empty chemicals in either of the two baths, you must first open the drain valve. After all liquid has run down the drain, close the valve(s).

Make sure to clean (rinse and wipe dry) the fume hood, all utensils and return everything to its place that you have used, after you are done.

Picture 15

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14.5 Sink



The sink uses regular tap water (not DI) and the drain goes directly to the public sewer (i.e. not connected to the pH neutralization process). This means **it is not allowed to empty any chemicals in the sink.**

No dirty utensils are allowed on the sink worktop. The sink worktop is intended for drying of washed and clean items.

All laboratory glass being in contact with substances which are not soluble in water (e.g. aluminum oxide or metal rests after lift- off process), should be carefully washed up by hand.

Do not put glass into the dishwasher if you have difficulty to remove any non- organic or organic rests. The dishwasher does not do a better job than you can do, and this may result in contamination of the machine.

14.6 Dishwasher

Most utensils, wafer holders, beakers etc. used in the Wet benches you just need to rinse with DI water.

Laboratory glass that have been used for solvents needs to be rinsed with acetone and/or isopropanol and then left to dry.

Put pre-cleaned laboratory glass into the machine and check that all pieces are stable. The water pressure can move items which may be damaged, so it is very important that they are placed properly on the pins. When the dishwasher is full, close the door and start the machine according to the instruction on the door.

Turn the label from "Dirty – load machine" to "Clean – unload machine". When the washing program is finished (the machine becomes silent) open the door, unload it and return all utensils to their place. Turn back the label to "Dirty – load machine".