

CLEAN WORK AND ITS CONSEQUENCES  
- Contamination Control Considerations -

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INTRODUCTION

The term "clean" by itself is indefinite in meaning and has different meanings in different contexts. It means different things to different people relative to their interests. "Clean" in terms of bacteria is not necessarily the same as "clean" in terms of chemical or particulate matter.

Cleanliness is related to the product considered; it is not an absolute measure. It is a relative condition denoting the degree to which a part or system may be cleansed of unwanted matter ( = contamination ). So the question to ask is not "How clean is clean? ", but "What is the contamination sensitivity of this part or system?". The area of attention is thus immediately directed from generalities to specifics.

If a mechanism or system must be free of contamination to function reliably , measures of contamination control must be taken. The purpose of any such effort is to reduce or manage contamination at the desired level. Biological contamination poses special problems because of the unique ability of living forms of matter to reproduce and grow.

Before implementing a contamination control program in a specific process, one should know the following:

- a). the type of contaminants which will be encountered
- b). the instance, when the contamination will occur (fabrication, subassembly, final assembly, testing , storage )
- c). the size, volume, mass and shape of each of these contaminants
- d). the effect of these characteristics on the ability of the product to tolerate contamination

In the case of superconducting cavities the level of attention brought to cleanliness may very well depend on the specific goals and may therefore vary from laboratory to laboratory. If e.g. the goal is to build a few meters of S - band cavities with fields of 3 MV/m and Q-values of  $3 \times 10^9$  it looks to me as a less difficult goal to achieve compared to the desire to build an

accelerator of several hundred meters with maybe 15 MV/m and Q-values of  $Q \geq 2 \times 10^{10}$  ( required by the higher gradients ) and maybe one can somewhat relax on the requirements for "care". Or it looks easier to protect a X-band cavity against external contamination from the environment than e.g. a 350 MHz resonator.

#### VULNERABILITY OF SUPERCONDUCTING CAVITIES TO CONTAMINATION

As we all know superconducting cavity devices are quite sensitive objects to deal with. Their characteristics like Q-value, break-down field and electron-loading behaviour is not always reproducible.

There is good evidence that contamination is the key to that behaviour; contamination can enter in the form of contamination of the bulk or contamination of the surface and we have heard already about these in previous talks.

Major contaminants of bulk niobium are interstitially dissolved gases or metallic impurities, which are acquired during the manufacturing process. In addition contaminants can be introduced into niobium during fabrication processes: stamping, spinning, machining, welding. Such gross contaminations are unacceptable and have to be avoided. In ref. 1 tungsten inclusions from TIG - welding have been identified as causes for break-down.

In 1970 my colleague O. Stoltz and I prepared our niobium cavities in that way that after the chemical treatment the cavity was rinsed in distilled water and methanol and dried with a nitrogen gas stream from a compressed gas cylinder before being assembled. When we changed our procedure to assembling the cavities wet and removed the methanol remaining on the surface by pumping out the cavity on the test stand, we experienced a pleasant surprise: the Q-value had improved by at least a factor of 5 to  $Q \sim 10^{10}$  and we were able to reproduce this result in subsequent tests. Obviously we had avoided contamination of the surface or at least reduced it.

Various groups have reported Q - degradations in cavities due to exposure in "hostile" environments (e.g. ref. 2 ) or due to vacuum accidents <sup>3)</sup>. It is generally believed that the observed Q - drops are caused by contamination of the superconducting surfaces with particulate matter.

During the developing phase of the KARLSRUHE-CERN separator the following observations had been made <sup>4)</sup>:

in several tests light emission from the cavity surface at localized spots had been observed at surface electric fields of 4 MV/m. Also severe multi-

pacting was experienced , which needed many hours to be processed.

An analysis of the emitted light from these sources led to the conclusion that this phenomenon was due to loosely attached dust particles at the surface. Similar light emission had been observed earlier in a 11 - cell muffin-tin cavity at CORNELL University<sup>2)</sup>. In a separate experiment the light emission could be studied and it was observed that the hot spots tended to sinter into the niobium surface and cooled down due to the improved heat sinking.<sup>5)</sup> Again it was concluded that loosely attached dust particles at the surface were heated up by the rf-fields. This conclusion was supported by the fact that tapping the cryostat generated more glowing spots at the surface. The increased light emission was accompanied with moderate Q - decreases and increased electronic activity.<sup>5)</sup>.

Chemical residues from surface treatments can cause break - down <sup>1)</sup>.

At CERN much reduced field emission was observed when cavities were dried and tested in a horizontal position<sup>3)</sup>.

To summarize: Contamination in the form of micro-particles on the surface, or chemical residue or foreign material inclusions have been identified to influence

- a). residual resistance
- b). electron loading ( multipacting , field emission)
- c). break - down fields

in superconducting cavities. Therefore it seems adequate to control the various forms of contamination:

## CLASSIFICATION OF CONTAMINATION

Contamination may be classified in 4 general categories :

- a). Particulates - dust, chips, fibers
- b). Chemical contamination - gases, liquids, inorganic compounds, organic compounds
- c). Biological contamination - bacteria, viruses, fungi, spores
- d). radioactive contamination

Each of these contaminants can be present in gases ( particularly air ), liquids ( particularly water ) or at surfaces.

Contaminants in liquids are:

- a). Non volatile residues like skin oils, solder fluxes, cutting fluids, cleaning solvents, plastizisers, lubricants...
- b). Other liquids in suspension or solution
- c). absorbed gases
- d). particulate matter
- e). viable and non-viable microbiological matter

In gases we find:

- a). Hydrocarbons ( residues from cleaning agents, lubricants)
- b). water
- c). absorbed or mixed-in gases
- d). particulates - corrosion, rust, flakes from wear ...
- e). microbial matter

Figure 1 shows some typical shapes of common particulate matter; figure 2 displays the sizes of some common contaminants .

In certain areas like the pharmaceutical or food industry microbiological matter is of special interest because of its ability to reproduce.

It is also of concern to microelectronics device manufacturers. The typical chemical constituents of living matter are <sup>7)</sup>: 77 % water , 15 % protein , 5 % fat , 2 % carbohydrate , 1 % other.

The quantity of microorganisms in different environments is given in table 1 :

Environment	Quantity
soil	$10^4$ to $10^6$ per gram
water	1 to $10^5$ per gram
air	10 to $10^4$ per cbft
human skin	1 to $10^4$ per sq. inch

Table 1 : Microorganisms in various environments <sup>7)</sup>.




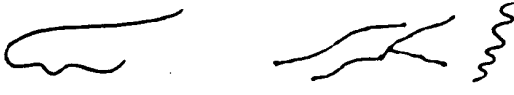

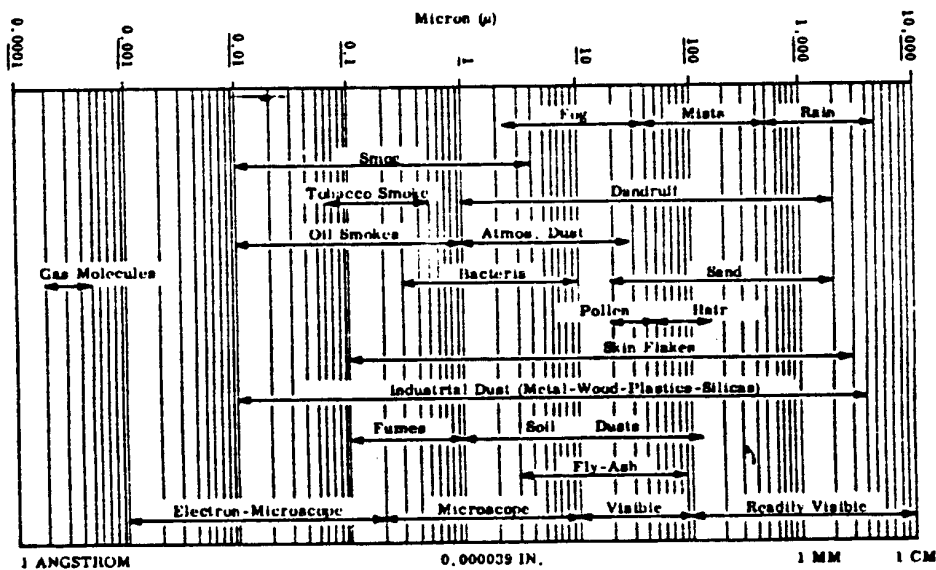
Shape	Appearance	Kind
Spherical		Vapor Pollen Fly Ash
Irregular or Crystalline		Mineral (Sand, Metallic, etc.) Cinder
Flake		Mineral Epidermis
Fibrous		Lint Plant Fibre Animal Fibre
Floc		Carbon Smoke Fumes

Figure 1 : Typical shapes of common particulate matter ( from ref. 6 )





 With this figure representing a particle 10 $\mu$  in diameter, the larger figure represents the cross section of the average human hair 100 $\mu$ . A major problem in contamination control is the tendency of the small particles to group and form larger particles.

Figure 2 : Approximate sizes of common particles ( from ref. 6 )

Table 2 summarizes the main groups of microorganisms :

Name	size	remarks
virus	10 to 300 nm	intracellular parasites, require living host cell
bacteria	< 1 $\mu$ m to 100 $\mu$ m	mobile, very effective reproduction by cell division, very resistant to adverse environmental conditions (spores)
fungi	5 $\mu$ m to 100 $\mu$ m $\emptyset$ ~ mm length	yeasts, mildews, mushrooms, molds
protozoa	1 $\mu$ m to several 100 $\mu$ m	found in water or animal bodies, very susceptible to drying
algae	~ 5 $\mu$ m to several meters	some kinds are very resistant to harsh environments

Table 2 : Main groups of microorganisms ( from ref. 7 )

If contaminants were immobile, many contamination problems would never exist. However, activity not only creates contamination, but also provides the mechanisms for moving contaminants from one place to another.

Contamination micration takes place as described in table 3 :

Carrier	Method of movement
Fluids	Contaminants are suspended, dissolved, adsorbed; particulates drop out as velocity decreases or as they become attached to surfaces
Solids	entrapped or adsorbed contaminants on solids move with it
Waves	heat, light, sound
Physical contact carrier	operator, tools, fixtures

Table 3 : Micration mechanisms of contamination

Contamination retention usually occurs through the attachment of particles, liquids or gases to surfaces. The more common mechanisms of retention are :

- a). Gravity
- b). Electrostatic charges - not only hold, but also attract contamination
- c). Molecular attraction - adhesion, adsorption, van der Waals forces
- d). Viscous surface coatings- common in machining, lubrication, skin oils
- e). Physical entrapment - rough or porous surfaces

CONTROL OF CONTAMINATION IN LIQUIDS AND GASES ( General principles )

Control of contamination in liquids uses several processes like settling, centrifugation, coagulation, filtration and ion-exchange. In gases filtration ( and absorption ) are the most common processes used for purification. Among those processes filtration and ion-exchange are the most important in "high technology" application .

FILTRATION is defined as the separation of solid particles from a fluid or gas by passing the mixture through a porous, fibrous or granular substance . The literature <sup>8)</sup> distinguishes between several kinds of filters and different filtration processes as summarized in table 4 :

Filter	feature	material	use
net or sieve filter	pore size $> 60 \mu\text{m}$ woven cloth	e.g. nylon	e.g. plankton filtration
depth filter	random array of overlapping fibers	paper, asbestos glass fibers	clarification -
membrane filter	porous structure depth filtration	cellulose acetat cellulose nitrat	prefiltration before membrane filters
ultrafilter	sieving of large molecules (molecular weight $10^5$ to $10^7$ ), defined cut-off	polymer film	ultrafiltration
RO - filter	molecular weight cut-off 50	polymer film	reverse osmosis

Table 4 : Filter types used in various filtration processes

Figure 3 illustrates the different structures of filters of various types.

Filtration is a rather complicated process. Particles suspended in a solution with sizes larger than the pore size of a filter are intercepted, but also particles smaller than the pore size may be retained due to adhesive or electrostatic forces. Most filters are negatively charged when wetted by a liquid with a pH - value  $> 2 - 3$ . Because cells, viruses and most macromolecules are also negatively charged, electrostatic repulsion forces can develop, which can overcompensate the adhesion forces and the particles are no longer retained in the filter. Special filters with positive charge at  $\text{pH} \approx 7$  have been developed and are generally used in virus work.

Filtration is a statistical process and if suspensions of a high enough density have to be filtered, some particles larger than the rated pore size of the filter can appear in the filtrate due to filter imperfections.

A filter must be wetted by the liquid to be filtered. Many filter materials like e.g. cellulose ester or teflon are hydrophob and must be wetted by filter agents. These can leach out from the filter and may contaminate the filtrate.

Gas - filtration is a completely different process than liquid filtration. In gas - filtration particles much smaller than the rated pore size will generally be retained. Whereas in liquid - filtration - because of the high viscosity of the fluid - only particle removal by interception is important, in gases inertial impaction and diffusion are of significance.

When a gas ( air ) ~~passes~~ through a filter, the velocity of flow increases as the gas passes through the filter pore according to Bernoulli's law. The stream

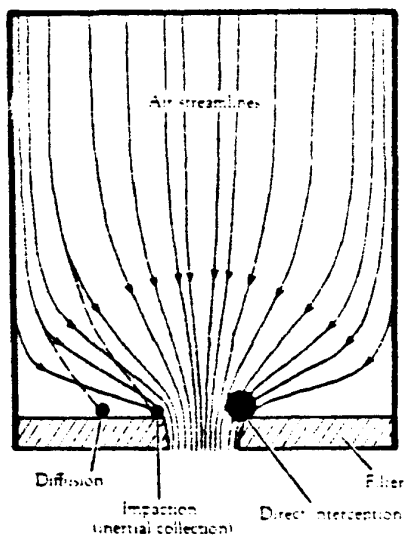


Figure 4: Principles of particle removal in an air stream

lines of the gas bend as they approach the pore (figure 4). Particles suspended in the gas behave differently than the gas itself and will be removed out of the gas stream by different processes:

a). removal by direct interception

occurs for particles larger than the pore size, but also for smaller particles, which have dimensions of an appreciably fraction of the pore size and therefore high probability of touching the filter surface.

b). removal by impaction

is the most important mechanism for removal of particles with diameters of  $\sim 1 \mu\text{m}$ . Because particles have inertia, they will tend to move along straight lines even if the stream-



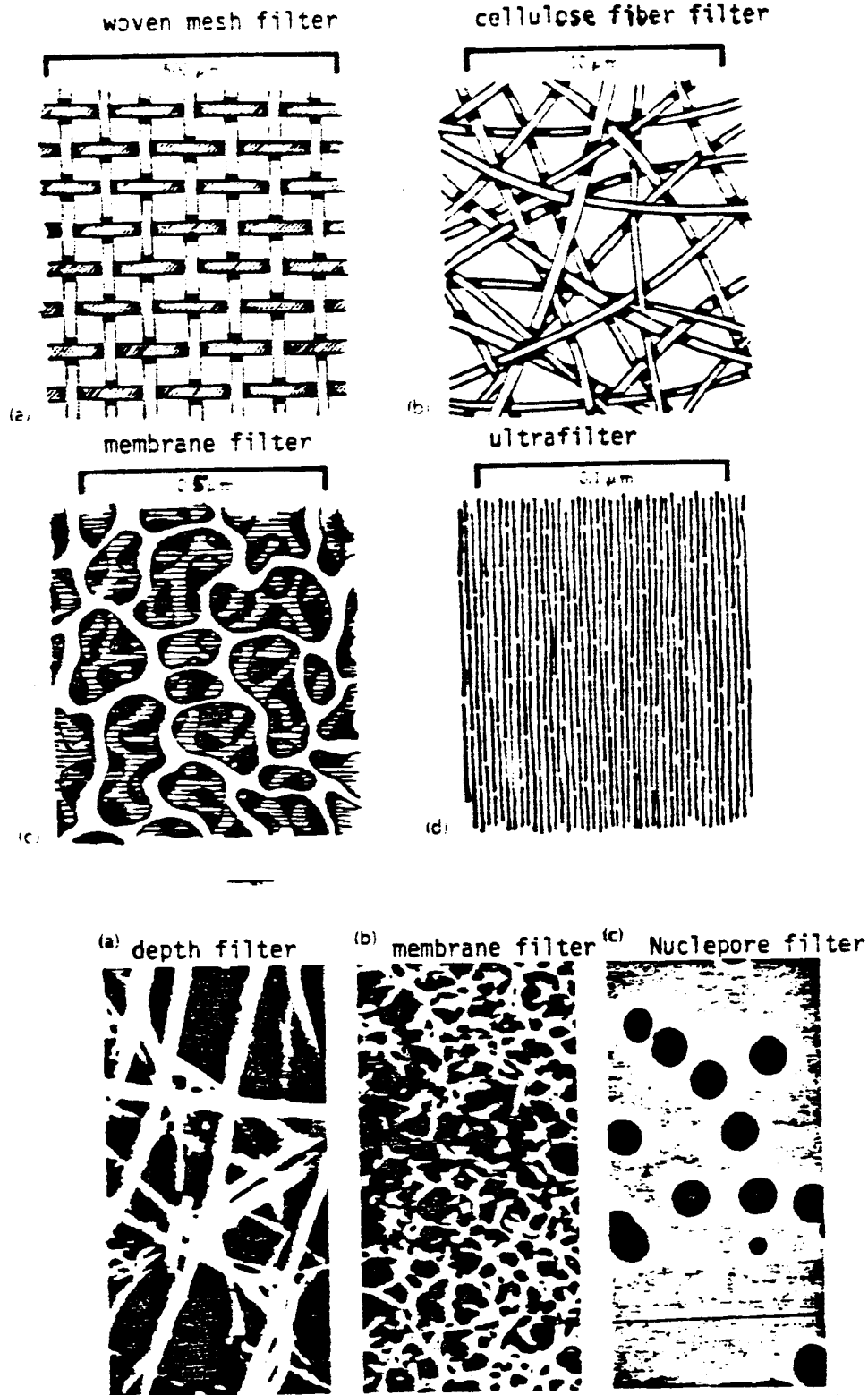


Figure 3 : Structures of filters of various types ( from ref. 8 )

of the air flow bend. Thus the particles impact the filter, when the stream lines bend near the filter pores.

Impaction is favoured by high gas velocities and dense filter matrices.

c). removal by diffusion

as a removal mechanism occurs only with very small particles ( $< 0.1 \mu\text{m}$ ), which exhibit Brownian movement and can diffuse in response to concentration gradients.

Since at the filter surface the particle concentration is originally zero, a concentration gradient is set up leading to a movement of particles out of the air stream onto the filter surface. This mechanism is favoured by low gas flow velocities and high concentration gradients.

d). removal by electrical attraction

- Filters can be negatively charged by normal manipulation or by the air flow itself. Thus positively charged particles can be removed via electrostatic attraction. This mechanism is effective for particles  $< 1 \mu\text{m}$ , but is generally considered to be of little practical significance.

e). removal by gravitational forces

is only important for very large particles and can generally be ignored in air or gas filtration.

ION -EXCHANGE is the process commonly used to remove unwanted dissolved inorganic contamination from water.

The ion - exchange process percolates water through bead like spherical resin materials, which exchange ions in the water for other ions fixed to the beads.

One distinguishes between 2 processes: a). softening and b). deionization.

Softeners contain beads that will exchange 2 sodium ions for every calcium - or magnesium ion removed from the "hard" water.

Deionization beads exchange either hydrogen ions for cations or hydroxyl ions for anions. The cation exchange resin, made out of cross-linked polystyrene and divinylbenzene, contains sulfonic acid groups and will exchange a hydrogen ion for any cation encountered ( $\text{Na}^+$ ,  $\text{Ca}^{++}$ ,  $\text{Al}^{+++}$ ).

Similarly the anion exchange resin, made out of cross-linked polystyrene and divinylbenzene containing ammonium groups, will exchange a hydroxyl ion for any anion (e.g.  $\text{Cl}^-$ ). The exchanged  $\text{H}^+$  - ions and  $\text{OH}^-$  - ions will form pure water.

Ion - exchange resins may be packaged in separate bed exchangers, with separate units for the cation and anion exchange beads, or in mixed bed exchangers, which contain a mixture of both types of resins.

CONTROL OF CONTAMINATION ( specific to superconducting rf-cavities)

Superconducting rf - cavities in their final stage of surface treatment are nowadays treated with

- a). acids
- b). water
- c). solvents

In the assembly stage they are in contact with gases or air.

In all these stages contamination takes place if not avoided or managed at a certain level.

Production of pure water

Water contains five basic groups of contaminants:

- a). dissolved inorganic salts ( Ca , Mg , Fe , Pb ....)
- b). dissolved organics ( herbicides , pesticides , detergents , polysaccharides...)
- c). gases
- d). suspended particles (pipe scale , dust , undissolved minerals and organics ...)
- e). microorganisms

All these contaminants have to be removed to a more or lesser degree depending on the specifications of the user.

Dissolved inorganics can be removed by ion - exchange units as discussed in the previous section. Dissolved organics can to some extent be removed by ion - exchange if they are ionic in nature. Non - ionic organics coat the ion - exchange resins and cause deterioration ( " fouling " ). Therefore they have to be removed from the water prior to ion - exchange , which is done by carbon adsorption.

This process is controlled by the diffusion rate of the organic molecules through the pores of activated carbon filters, and by the pore diameter. The rate of adsorption is a function of the molecular weight and molecular size of the organics.

Suspended particles and microorganisms can be removed by reverse osmosis (RO) appropriate filters and ultraviolet sterilization. Figure 5 explains the principle of RO: OSMOSIS is molecular diffusion of the solvent from the less concentrated solution to the more concentrated solution through a semi-permeable membrane.

REVERSE OSMOSIS is the movement of the solvent ( water ) through a membrane in the opposite direction as in osmosis and this happens only under the influence of an applied pressure. It is a process to reduce the solute concentrations , in this case to increase the purity of the water.

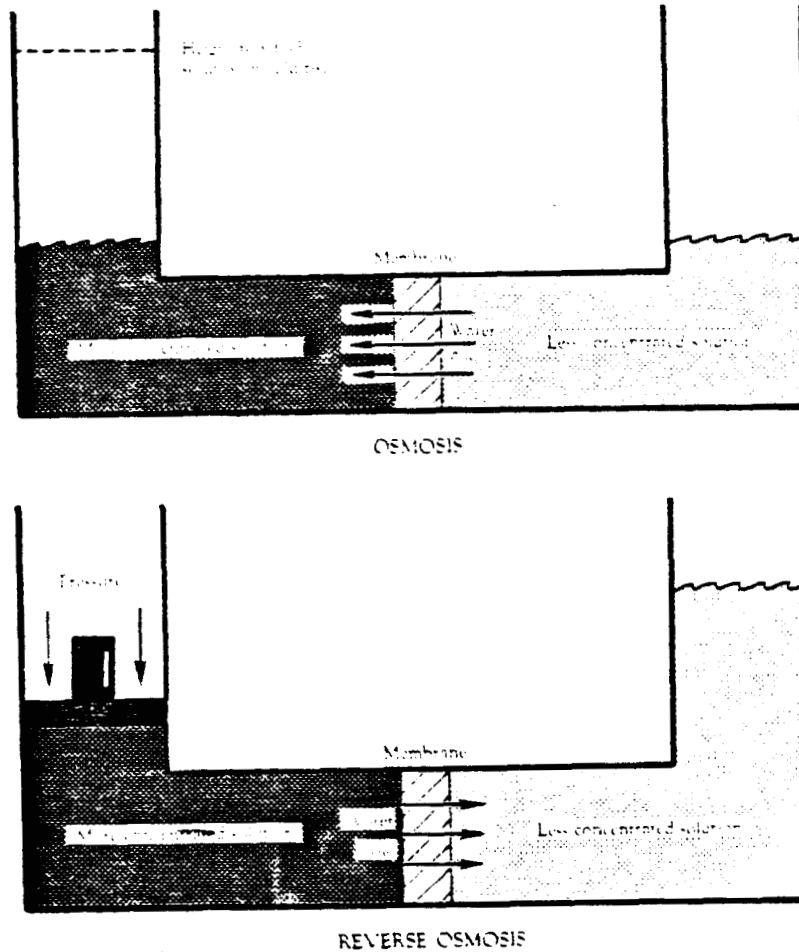


Figure 5: Principle of reverse osmosis

The obtained water quality in a water purification system is judged by 4 independent parameters :

- a). resistivity
- b). particle content
- c). total organic carbon content ( TOC )
- d). viable bacterial count

Microelectronic device manufacturers have recognized that all of these parameters are important for product quality and fabrication yield. The vast majority of water related problems can be linked to either of these parameters <sup>9)</sup>.

Bacterial contamination represents one of the most serious threats to ultra-pure water systems. As mentioned earlier, bacteria are self-generating, non-ionizable particulates, which can adapt their metabolic processes to harsh environmental conditions. 18 MegOhm cm water with TOC levels of 20  $\mu\text{g/l}$  still can contain  $10^6$  bacteria per ml <sup>10)</sup>. Therefore special construction principles have to be followed: one has to avoid any areas of stagnant water in an ultra-pure water system. Threaded or welded socket type joints leave cracks, which promote bacterial growth. Butt-welded heat fusion joints have to be done.

Ball valves, the most common used flow control device in a pure water system, are sources of major dead fluid areas and promote bacterial growth <sup>11)</sup>. Diaphragm valves, which have only 2 wetted surfaces and no dead space, are the solution to this problem. Each point of use of ultra-pure water should be protected with appropriate filters against bacterial contamination from the environment.

Ultra-pure water is an excellent solvent: it can pick-up contamination from any environment it encounters. That means that piping and process vessels or storage tanks are very vulnerable, if the wrong material is used for construction. PVC leaches significant quantities of organics <sup>12)</sup>. Unpigmented polyvinylidene fluoride, which lacks plasticizers, is the recommended material <sup>13)</sup>. Liners in ion-exchange vessels as well as in storage tanks can be sources of contamination and leach out plasticizers. Generally fluoropolymer linings are not attacked by ultra-pure water. Even ion-exchange resins can be additional sources of contamination. Therefore "Nuclear grade" resins, which are treated to reduce organic extractables, are recommended for the polishing deionization system <sup>14)</sup>.

Levels of particulate matter in ultra-pure water systems used by microelectronic device manufacturers are 1 - 2 particles per ml. For comparison, the water in the Cornell system contains 50 to 100 particles/ml. In test filtrations with different filter types we were able to reduce the particle content to 4 particles/ml.

Control of the above mentioned quality parameters in an ultra-pure water system is essential but not always easy. Resistivity and particle count can be monitored with resistivity sensors and particle counters using a light scattering principle. For bacterial counts kits with filters and nutrients, which grow bacterial colonies in a given incubation time and from which the original number of bacteria can be calculated, are available. A more direct method is epifluorescence microscopy <sup>15)</sup>.

Most crucial apparently is the determination of the TOC-level, because it is very sensitive to contamination by the sample collection process. Also the base-line calibration is difficult, because it needs a reliable source of organic free water <sup>16)</sup>. The analytical methods used are either ultraviolet oxidation resistivity detection or wet oxidation or chromatographic methods.

Figure 6 shows an example of an industrial ultra-pure water system used by a major computer component manufacturer. The system produces water of 18 MegOhm cm, with 2 particles/ml of  $>1\mu\text{m}$ , 0.01 viable microorganisms/ml and a TOC - level 200  $\mu\text{gr}/\text{ml}$ .

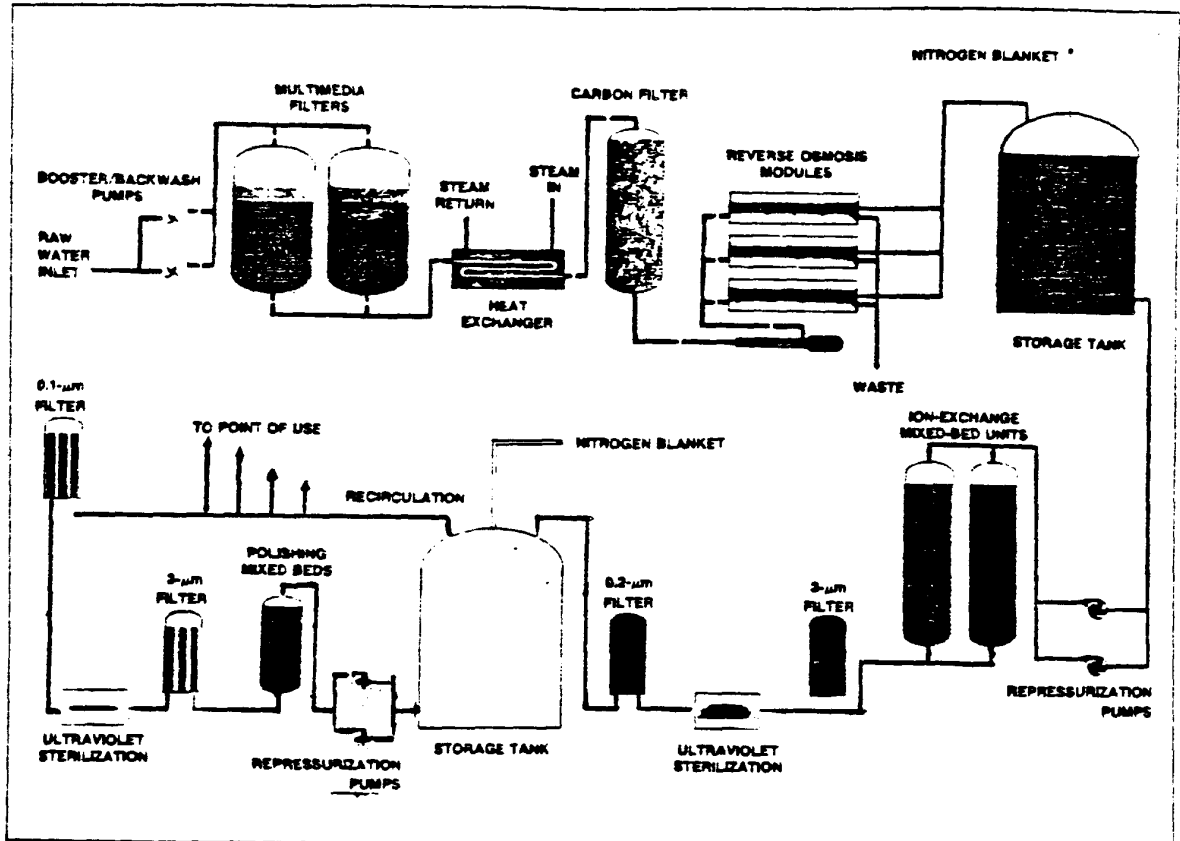


Figure 6 : Example of an industrial ultra-pure water system (from ref.13 )

### Chemicals

Initially the semiconductor industry placed much emphasis on the quality of the water used in the rinsing stages of the chemical processes involved in wafer production. Nevertheless it was recognized that the philosophy that ultra - pure water will remove all traces of chemicals is only true for impurities soluble in water , but is not valid for insoluble constituents. Such particulate contaminants in the processing chemicals for etching and rinsing can cause severe malfunctioning of integrated circuitry and as an example the presence of only 2 ppm of sodium at the  $\text{SiO}_2/\text{Si}$  interface of a MOS - device can cause large changes in threshold voltages at gates <sup>17)</sup>.

Investigations have shown that considerable quantities of particulate matter

are present in electronic grade chemicals , which can only be controlled by ultrafiltration techniques prior to the application and not by pure water rinsing.

Table 5 contains the results of an investigation by D.Lafeuille et al. <sup>17)</sup>, who used as analytical methods neutron activation ,flameless atomic absorption and X-ray fluorescence :

Chemical reagent	Grade	total impurity[ $\mu\text{g} / \text{l}$ ]		
		Na	Cl	Mn
70% $\text{HNO}_3$	reagent	450	120	0.3
	semiconductor	120	3300	0.1
49% HF	semiconductor	70	2400	16 - 180
30% $\text{H}_2\text{O}_2$	semiconductor	17	60	0.02

Table 5 : Impurity levels in selected chemicals ( ref.17)

The particulate content in buffered hydro-fluoric acid consisted of  $160 \mu\text{g}/\text{l}$  Fe ,  $200 \mu\text{g}/\text{l}$  Ca ,  $55 \mu\text{g}/\text{l}$  Zn,  $11 \mu\text{g}/\text{l}$  Al ,  $10 \mu\text{g}/\text{l}$  Pb as the major impurities. It was also noted that the impurity levels varied from batch to batch from the same supplier.

With the fast development of VLSI - circuitry and the shrinking dimensions of individual components ( $\approx 3 \mu\text{m}$ ) the need for controlling particulate matter in chemicals has increased further <sup>18)</sup>. Juleff et al. <sup>18)</sup> report about investigations of various chemicals filtered to  $0.2 \mu\text{m}$  in a class 100 clean room (table 6 ) :

Chemical	particle count by SEM
Isoclean acetone	} $\leq 4$ particles / ml of $d \geq 3 \mu\text{m}$
Isoclean methanol	
Isoclean sulfuric acid	
Isoclean hydrofluoric acid	

Table 6 : Particle content of high purity chemicals ( from ref. 18 )

For comparison the chemicals used at Cornell for the chemical processing are compiled in table 7 :

Chemical	Grade	residue after evaporation	particles/ml	supplier
70% HNO <sub>3</sub>	electronic	0.0004%	-	Hi-pure Chemical
85% H <sub>3</sub> PO <sub>4</sub>	electronic	0.005%	-	Ashland
48% HF	analytical	0.0005%	-	Malincrodt
30% H <sub>2</sub> O <sub>2</sub>	reagent	0.002%	-	Ashland
Acetone	reagent	0.001%	100	Ashland
Methanol	multi-purpose		100	Burdick+Jackson

Table 7 : Chemicals used in cavity processing at Cornell ( LNS )

As indicated in the table most of these chemicals leave significant residues after evaporation. Figure 6 shows some typical stains produced from various solvents ( from ref.18)

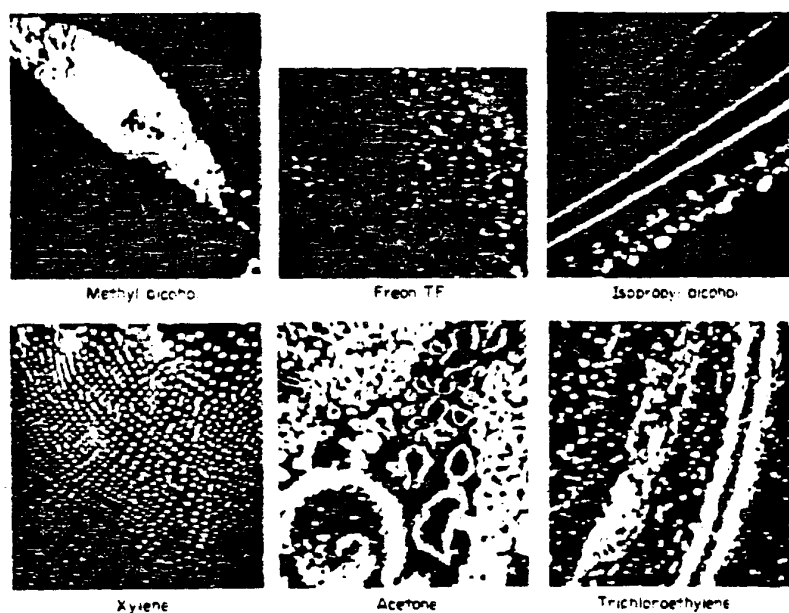


Figure 7 : Solvent stains ( dark field optical microscopy )

Ultra-violet/ Ozone cleaning devices are on the market, which seem to be especially suited for removing hydrocarbon contamination via complete oxydation <sup>19), 32), 33)</sup>.



By comparing tables 5,6 and 7 one can see that improvements in the purity of chemicals are possible if necessary, which is a question open to discussion. One might argue that at the present time the most important areas in superconducting rf- cavity development are residual resistance and field emission loading. One might also speculate that field emission is more susceptible to point - like contamination by particulates than residual resistance ( at the level of surface cleanliness achieved nowadays interface losses <sup>20)</sup>. seem to be more important ). The fact that field enhancement factors around or below 100 (  $\beta \times E \approx 5000 - 7000 \text{ MV/m}$  ) have been obtained <sup>21)- 24)</sup>. suggests that the normally encountered field enhancement factors of  $\beta \approx 250 - 800$  are not an intrinsic feature of the material itself ( dielectric inclusions <sup>25)</sup>. as e.g. observed in mechanically polished field emission electrodes are less likely because of the different surface preparation of niobium ). Field emitted electrons accelerated in rf - fields deposit power at the cavity wall proportional to  $\exp(K/\beta E)$  and only a certain power dissipation can be tolerated by the material, or in the emitter . By reducing the enhancement factor from typically 250 to 100 , which might or might not be possible by maintaining good control of surface contaminants, it could be possible to increase the fields by a factor of 2.5 , assuming that no limitations by thermal runaway occur.

Such measures of contamination control as discussed so far are only useful ,if the level of surface cleanliness can be maintained during the assembly and test stages. An usual practice of supposedly protecting clean parts like flanges, couplers, rf - probes , which have to be mounted onto the cavity, from contamination is to wrap them into aluminum foil. Also the cavity ports are often closed with foil. According to the literature <sup>7)</sup>. aluminum foil is one of the worst materials to use for maintaining cleanliness of a surface because of excessive sloughing. Table 8 contains some materials to be considered for "packaging" together with their advantages and disadvantages :

Material	Comment
Aluminum foil	<ul style="list-style-type: none"> <li>a). household foil has oil film on surface</li> <li>b). high rate of particle release ("sloughing") due to abrasion and flexing</li> <li>c). surface oxides can be abrasive to delicate surfaces</li> <li>d). contact with dissimilar metals can contribute to corrosive action</li> </ul>
Polyethylene foil	<ul style="list-style-type: none"> <li>a). not recommended for critical cleanliness levels, sloughing of hydrocarbons</li> <li>b). available as antistatic and daylight fluorescent film for tracer techniques</li> </ul>

Material	Comment
Polyamide Nylon - 6 film	a). recommended as packaging material for items requiring the <u>ultimate in particulate cleanliness</u> ; high abrasion and flex-resistance b). available as antistatic and daylight fluorescent film

Table 8 : Selction of materials used for "packaging"

Assembly of a cavity is usually done in a controlled, contamination free "clean room " and I shortly want to discuss this facility in the following .

Control of airborne contamination ( "clean room " )

Air contains organic and inorganic gases as well as aerosols (dust, smoke, fumes, ash, soot, oil mists, liquid droplets ...). The only effective control of airborne contamination is achieved by laminar air flow , "airflow in which the entire body of air within a confined area moves with uniform velocity along parallel lines". A confined area, which has this capability , is called a "Laminar Air Flow Clean Room". It has the following features:

- a). a self- clean-down capability to combat both contamination brought into the room or generated within the room.
- b). an air flow pattern ~~which~~ carries the airborne contamination away from the work and the work area.

The cleanliness of the room is achieved by forcing large quantities of air through "High Efficiency Particulate Air Filters " ( HEPA ), which are made out of glass/asbestos fiber assemblies with fibers  $\leq 1 \mu\text{m}$  in diameter. The efficiency of such filters is minimal 99.97 % for particles  $> 0.3 \mu\text{m}$ . The airflow through the filters has to be adjusted to the size and configuration of the room.

Clean rooms are characterized by their degree of cleanliness as shown in table 9 :

Class	maximum number of particles $> 0.5 \mu\text{m}$ per cbft	maximum number of particles $> 5 \mu\text{m}$ per cbft
100	100	not defined
10000	10000	65
100000	100000	650

Table 9 : Classifications of clean rooms

For comparison table 10 contains some particle concentrations in different areas :

Area	concentration per cbft
rural	100 000
city	1000 000
chem.room	100 000
experimental hall	210 000

Table 10 : Particle concentrations in selected areas

Vertical clean rooms will very well operate in the class 100 level with air velocities of  $\sim 30$  m/min. The basic advantage over horizontal clean rooms is the non - existence of cross - contamination between adjacent operations.

Horizontal clean rooms are always " zoned " : close to the filter bank a class 100 environment exists, but operator and operation in this zone influences the cleanliness of the zones further away.

One of the largest emission sources in a clean room is the personal. The particle emission of a normally dressed person , which is not moving , is  $10^5$  particles / min; violent movements increase this level by a factor of 100.

Special garmenting ( sufficient impenetrable fabric weave ) has to be used , which has the purpose of

- a). containing as much of the particulate matter brought into the area
- b). also contain the particles generated by the person during the time the garment is worn .

To achieve and maintain the cleanliness level of a clean room ( assuming the proper construction material is used ) the appropriate equipment has to be used like : benches and work-surfaces without drawers, braces, footrests and with beveled edges, appropriate cabinets, chairs, stools , tools , including lint-free towels and paper. It is also good practice to monitor the cleanliness level of a clean room with particle monitors; one immediately recognizes degradation and can take appropriate steps.

In the semiconductor industry and in the field of antibiotic fermenting, where air sterilization is essential, teflon membrane filter cartridges with the ability to quantitatively remove airborne particles as small as  $0.02 \mu\text{m}$  are used with high flow rates ( 1200 scfm ). Smaller versions of such filters are already in use for ultrafine filtration of electronics process gases, including air. There are

trends to replace dry filtered air ( e.g. with activated charcoal filters in tandem with HEPA - filters) in favour of an inert gas like nitrogen in areas, where inert quality is necessary.

Because of the fact that the operator is one of the major emission sources in a clean room the industry has developed certain patterns in personal management. It is considered most important for a successful contamination program to achieve good employee participation .The elements for that are

- a). a knowledge of what must be done and how it can be best accomplished
- b). an understanding or awareness of the necessity for contamination control and the result of failing to do so
- c). the creation of a receptive and cooperative attitude

#### Further sources of contamination of a cavity

During the testing stage the cavity is subject to contamination and I have mentioned this fact in my introductory remarks. Ideally one would like to separate the cavity interior totally from the exterior elements of the test set-up like pumping lines, tuners or coupling lines. This has been practiced with good success <sup>26)</sup> at Stanford University on test cavities. Nevertheless in several cases there was the suspicion of contamination from e.g. rf - windows because of the fact that the break-down was usually observed at the bottom of a cavity.

Unfortunately closed cavity techniques are not always applicable or only with great efforts , especially for larger cavity assemblies. Contamination of this kind ( from feed lines , vacuum system ) can to some extent be avoided by proper designs like coupling and pumping from below, horizontal testing ...

One might ask the question in what stage of the fabrication and testing process of a superconducting cavity contamination control measures have to be taken. Obviously a good attitude would be to try to limit contamination to a certain degree from the very beginning of the fabrication process, because it might be difficult in later stages to remove the contaminants or they might be overlooked in the quality control process. As trivial as it sounds :

Once the niobium is accepted as useable, the cutting , stamping, spinning or machining process should neither introduce foreign material nor mechanical damage like scratches, gauges etc. If annealing stages are included in the manufacturing process the parts should be carefully cleaned from particulates, organic or inorganic matter. This is not always simple if one considers that the forces of adhesion, which hold particles at surfaces, are quite high. For example

a particle of 50  $\mu$ m size is held at the hood of a motor car with a force, which requires a speed of nearly 200 km/h to be overcome. Appropriate cleaning techniques like ultrasonic agitation and flush cleaning have to be applied in conjunction with the appropriate cleaning agents. In table 11 some features of a few selected cleaning media are summarized :

cleaning medium	removes org. liquids	org. solids	dissolved inorg. solids	inorg. liquid silicon oil	removes particulates	purity	chem. stability
Methanol	yes	yes	limited	yes	fair	fair	fair
Acetone	yes	yes	no	yes	poor	good	good
DI-water	no	no	yes	no	fair	very good	excellent
trichloro-trifluoro-ethene	yes	fair	no	yes	very good	excellent	excellent

Table 11 : Selected list of cleaning agents ( from ref.7 )

Another area which needs high attention is the electron beam welding. Not only must the parts to be joined have good mechanical tolerances, but also they must be free of chemical residues or particulates. At Cornell we experienced many set-backs in our welding procedures, which could be partially tracked back to contamination. Contaminants trapped in the welding steps tend to evaporate when hit with the electron-beam and cause splatter of the molten niobium, resulting often in holes. We find it essential to inspect the parts to be welded under a microscope prior to putting them together.

WHAT INFLUENCE HAS CONTAMINATION CONTROL ON CAVITY PERFORMANCE - EXPERIENCE

In the final part of this contribution I want to address the question "What has been gained by the "clean work" methods applied at this point to rf-cavities?"

Our presently applied methods are more or less limited to

- a). the use of 18 MegOhm cm water for rinsing
- b). the use of laminar flow clean rooms or benches for assembly
- c). the application of appropriate testing arrangements like horizontal placement of the cavity in the cryostat or pumping and coupling from below

The general experience seems to be that no dramatic improvements in cavity performance

has occurred by implementing the above measures. Nevertheless the experimental results obtained with the cavity treatment processes of to-day ( wet - treatment ) , which have for quite some time lacked behind the "furnace" treatments of the 70's, have caught up or surpassed these results. Ten years ago a typical L-band cavity measured at Stanford University after ultra-high vacuum firing exhibited Q - values of  $Q > 10^{10}$  and surface electric fields of 16 - 20 MV/m.<sup>27)</sup>.

In multi-cell S - band cavities accelerating gradients of up to 6.5 MV/m and Q - values of  $10^{10}$  have been achieved<sup>28)</sup>. As reported in ref. 29 , such cavity performance has been reproduced in the last years with to-days technologies. As we have heard<sup>30)</sup>, the gap between test results on single cell cavities and actual accelerating devices is closing .

Field emission loading remains the major factor troubling superconducting cavities, but some progress has been made by appropriate assembly<sup>3)</sup>. There are more often measurements reported, which show little field emission or smaller field enhancement factors.

Results have become more reproducible by applying the above mentioned techniques and this seems to be a very important experience, if one thinks about building larger cavity assemblies. It is difficult to imagine that for a superconducting LEP or TRISTAN - ring each individual cavity section has to be brought up to the design values of Q and field by successive laboratory tests.

As I have tried to discuss we are not applying contamination control measures in full consequence and we are still far behind in our efforts when compared to the developments in the microelectronics industry. One can question at this point the necessity for more stringent control measures, but it might be a valuable exercise to try to apply the state-of-the-art technology in contamination control to a smaller cavity. More stringent contamination control measures might be necessary for large scale cavity fabrication and it might be essential to reduce manual handling in the processing steps of the surface treatment whenever possible as it is done already in the microelectronics circuitry production<sup>31)</sup>, because the operator is one of the largest sources of contamination.

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