

Particle Monitoring in Liquids



Agenda



- **Special considerations for liquids**
- **Types of liquid particle counting systems**
- **DI water systems**
- **Chemical manufacturing and management systems**
- **Point-of-use wet process monitoring**

Special Considerations For Liquids



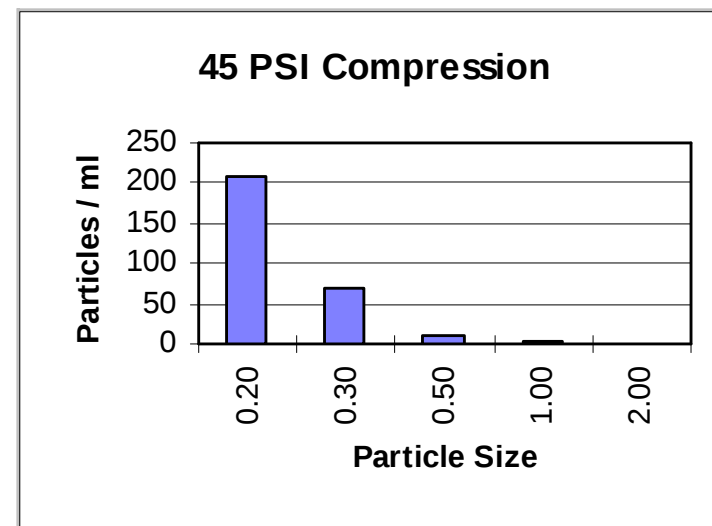
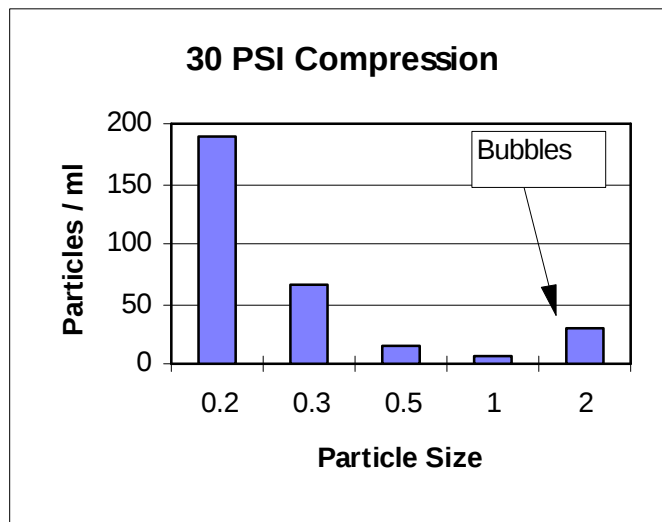
- **Bubble formation**
- **Index of refraction**
- **Particle counting coincidence**
- **Contamination**



Bubble Formation



- Bubbles are counted as particles
- Many process chemistries form bubbles readily
- Pressure can often return bubbles to solution

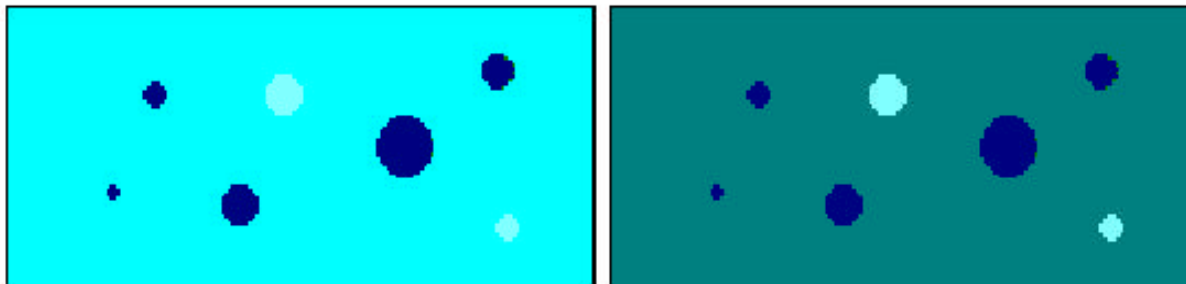


Index of Refraction



- **Optical particle counters rely on index of refraction contrasts**
 - Sample media v/s particle
- **Contrast variations impact sizing accuracy**

$$IR_Contrast = \frac{Particulate_IR}{Media_IR}$$



Index Of Refraction



▪ Refractive Indices of Semiconductor Materials

Particulate Material	Refractive Index
Silicon Dioxide	1.45
Silicon	3.90
Polystyrene Latex	1.59
Sample Media	
Hydrofluoric Acid (50%)	1.29
Ammonium Hydroxide (29%)	1.33
Hydrochloric Acid (37%)	1.41
Sulfuric Acid (96%)	1.46
Water	1.33

Index of Refraction Effects

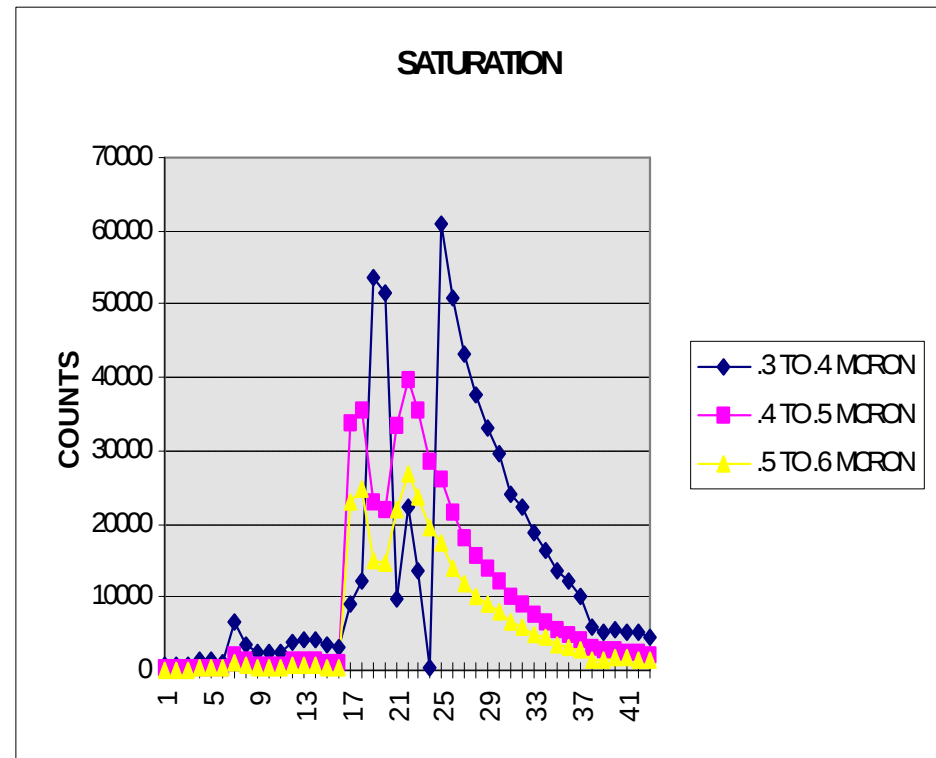


- **Particles of like material with equivalent light scatter (780 nm) in various liquid media**

H₂O, NH₄OH	HF	H₂O₂	HCL	H₂SO₄
0.3	0.28	0.31	0.36	0.40
0.5	0.46	0.52	0.62	0.72
1.0	0.87	1.10	1.80	2.30
5.0	5.20	5.00	6.00	7.90

Particle Counting Coincidence

- High particle concentrations overwhelm particle counters
- Optical coincidence = more than one particle in the viewing region at once
- Multiple particles size as one larger particle
- Particle coincidence is indicated by a perceived size distribution shift



Contamination



- **Particles and other residue can contaminate the wetted surfaces of a liquid particle counter**
 - Optical surfaces
 - Sample lines, fittings and valves
- **Volumetric sensors are more sensitive to contamination build-up**
- **Contamination will affect cleanup rates and baseline particle counts**
- **Often identified by:**
 - DC Light level changes
 - Increased concentrations in first sizing channel

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- **Types of liquid particle counting systems**
- DI water systems
- Part cleanliness testing
- Chemical manufacturing and management systems
- Point-of-use wet process monitoring

Volumetric Liquid Particle Counter

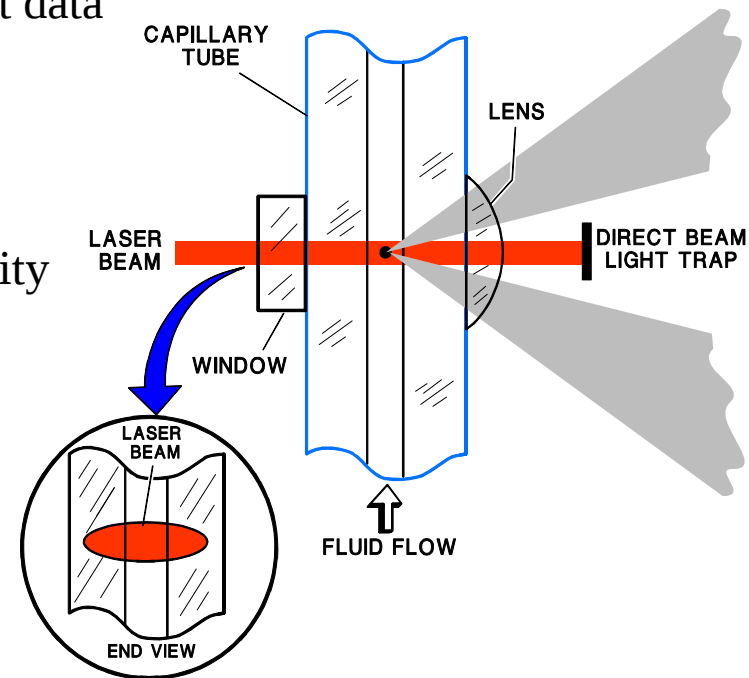


■ Pros

- 100% of fluid flow analyzed for particle count and size
- More repeatable results instrument to instrument
- Reduced time to statistically significant data
- Uses less sample fluid

■ Cons

- More expensive for equivalent sensitivity
- More sensitive to contamination
- Greater pressure drop



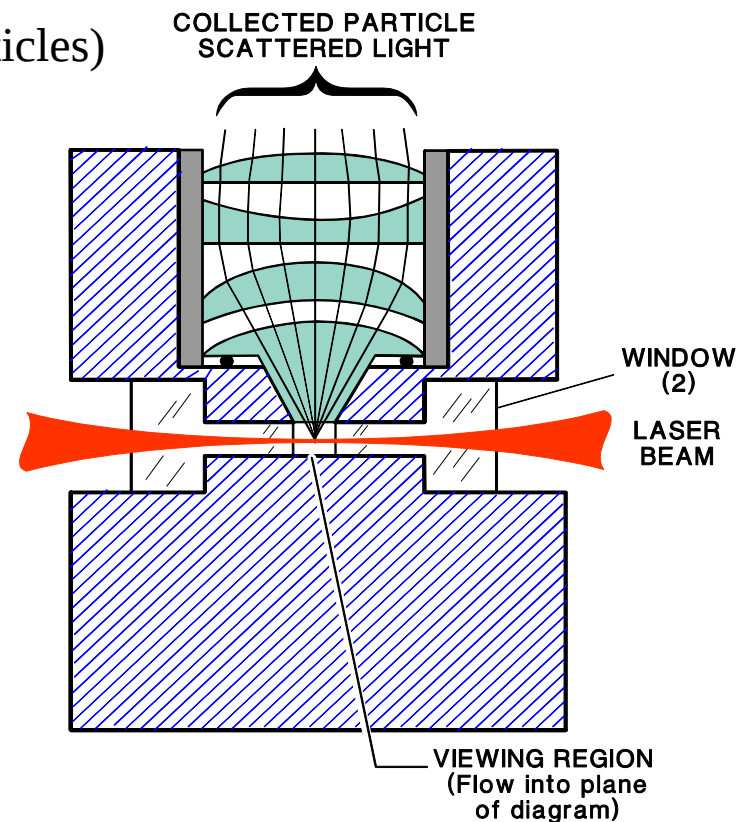
Non-Volumetric Liquid Particle Counters

■ Pros

- Most sizing sensitivity (sees smallest particles)
- Less expensive for equivalent sensitivity
- Less sensitive to contamination
- Less pressure drop

■ Cons

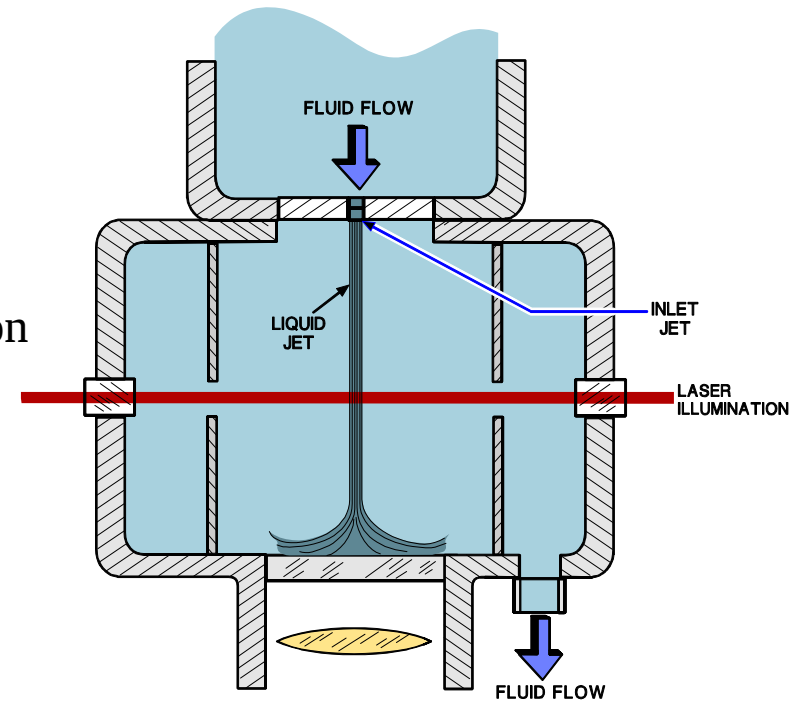
- Small portion of total flow analyzed
- Inconsistent sample volume
- Longer time to statistical significance



Inviscid Jet Liquid Particle Counters



- **New Technology**
 - Not a capillary design
 - Measure a jet of chemical
- **Pros**
 - Volumetric
 - Good sizing sensitivity
 - Little sensitivity to optical contamination
- **Cons**
 - Fair size resolution
 - More expensive than non-volumetric

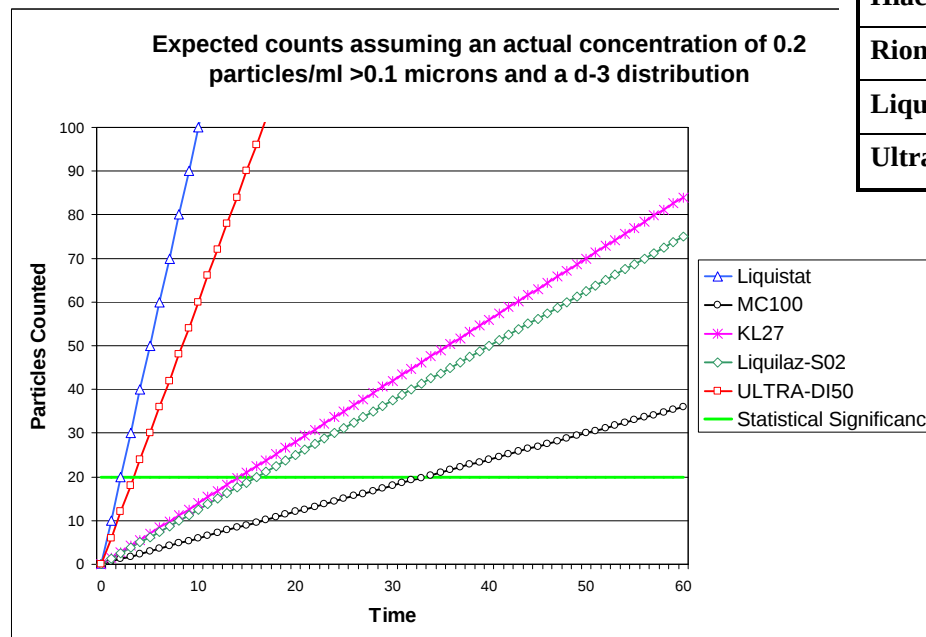


Sample Volume vs Sizing Sensitivity



- Sample volume is often more important than size sensitivity
- No filtered system is perfect
- Distributions follow predictable patterns
- Key measure is total particles counted

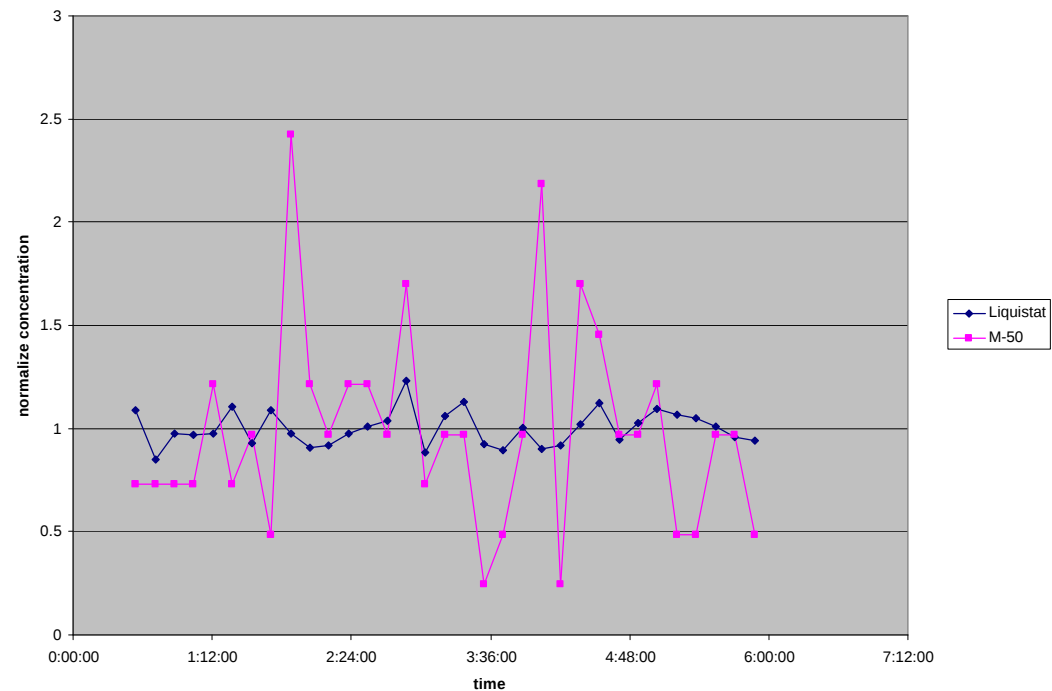
Sensor	Time to statistical significance (min)	Vol. to statistical significance (ml)
Liquistat	2.00	100.00
Hiac MC100	33.33	3333.33
Rion KL27	14.28	142.85
Liquilaz-S02	16.00	800.00
Ultra-DI50	3.33	3333.33



Particle Count Rate Effect on Data Variability



- **Increased statistical significance**
 - Reduced inter-sample standard deviation
 - Allows tighter QA controls



Volume Effects on Statistical Control Limits

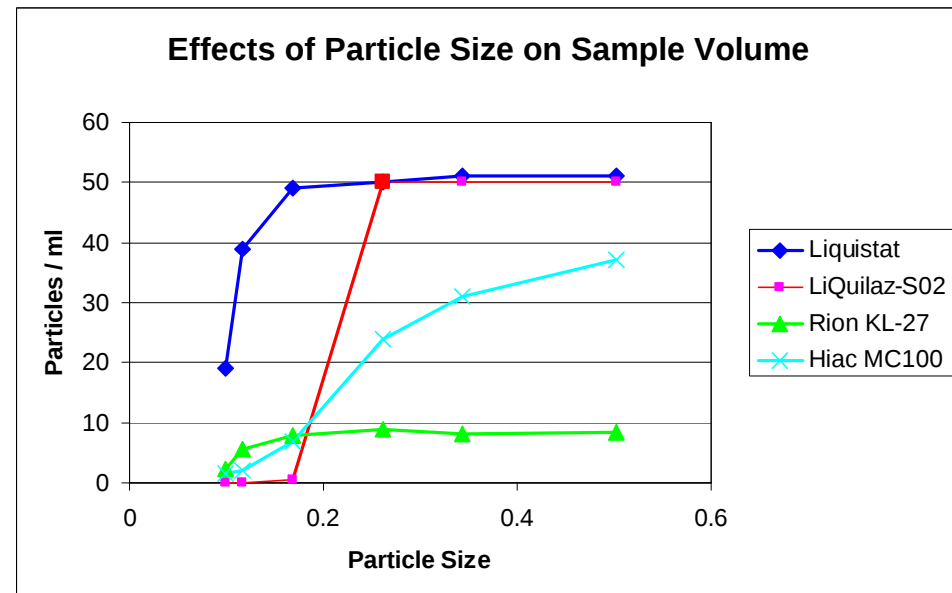


- **Given**
 - A particle control specification at $0.8 / \text{ml} > 0.2 \text{ microns}$
 - An average particle concentration of $0.4 / \text{ml} > 0.2 \text{ microns}$
 - Sensors taking 10 minute samples over 500 minutes
 - HSLIS-M65 ($0.065\mu @ 0.6 \text{ ml/minute assay}$)
 - Liquistat-100 ($0.1\mu @ 50 \text{ ml/minute assay}$)
- **HSLIS-M65 expected = $((10_{\text{min}} * 0.6_{\text{ml}}) * 0.4_{\text{particle/ml}}) = 2.4_{\text{particles / sample}}$**
- **Liquistat-100 expected = $((10_{\text{min}} * 50_{\text{ml}}) * 0.4_{\text{particle/ml}}) = 200_{\text{particles / sample}}$**
- **95% UCL per sample = (expected counts) + $1.65 * \text{SD}$**
 - HSLIS = $2.4 + 1.65 * 1.55 = 4.96 \text{ particles per sample} = 0.83 / \text{ml}$
 - Liquistat = $200 + 1.65 * 14.14 = 223.331 \text{ particles per sample} = 0.45 / \text{ml}$

Effects Of Inconsistent Sample Volume



- Particle counter design dependent
 - Normally a problem with non-volumetric designs
 - Measured volume per unit time changes with size



In-line Particle Counter Systems



- **In recirculation or delivery systems**

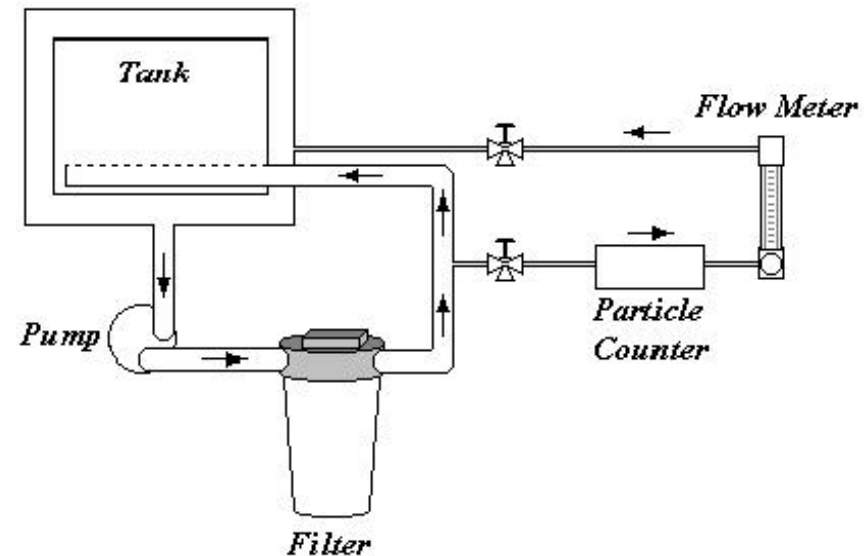
- Downstream of pump
- Downstream of filter

- **Pros**

- Smaller size
- Continuous sampling
- Lower price per sample point
- Less susceptible to contamination

- **Cons**

- Possible bubble problems
- Requires plumbing modification



Compression Particle Counter Systems

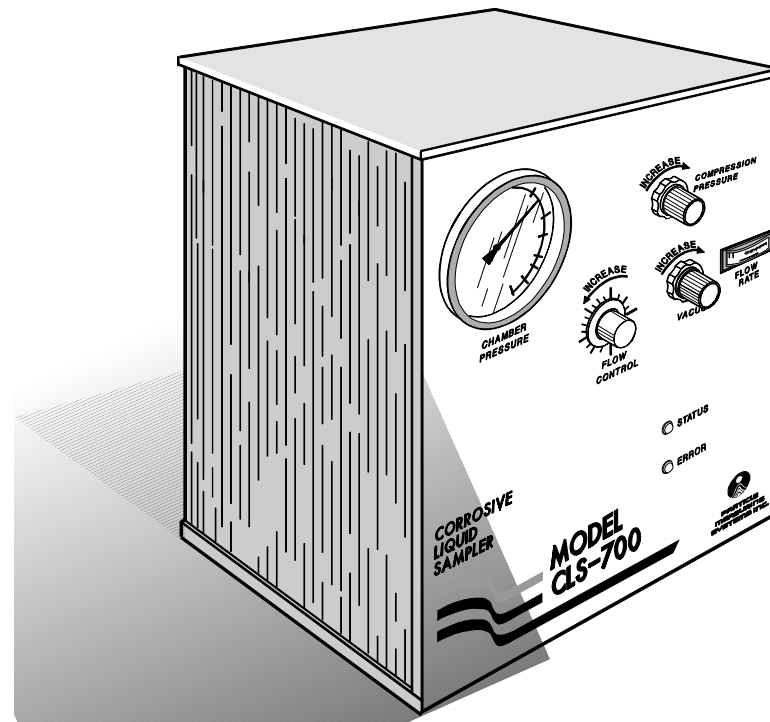


■ Pros

- High temperature compatible
- Measure effervescent fluids
- Sample triggering

■ Cons

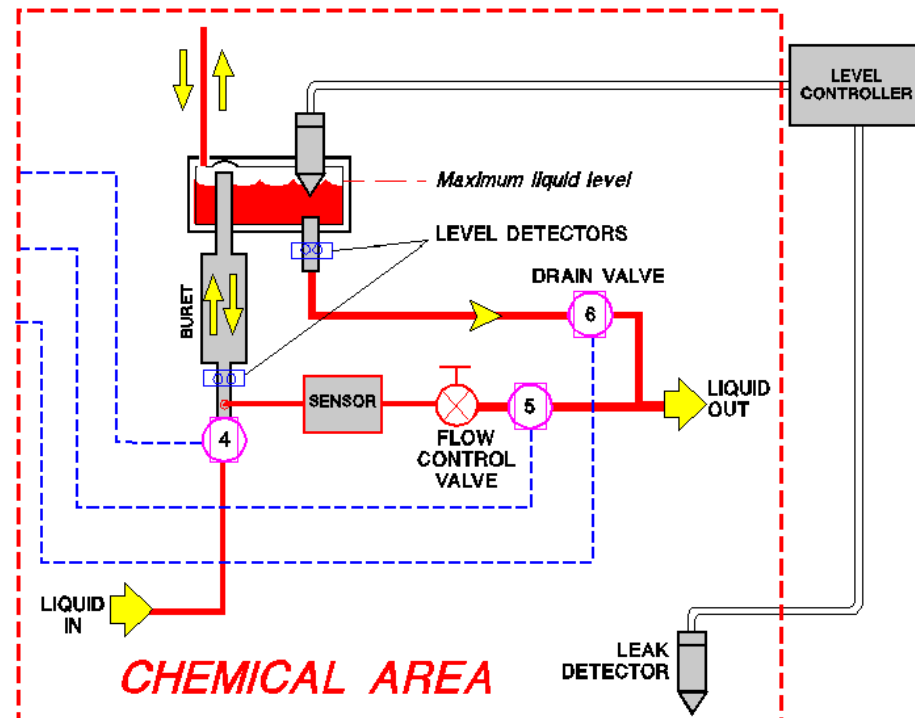
- Higher price per point
- Not continuous
- More susceptible to contamination



Compression Particle Counter Operation



- Draw sample through buret and into overflow chamber
- Discard overflow fluid
- Compress sample to eliminate bubbles
- Force sample through particle counter
 - Constant pressure
 - Constant flow rate



Syringe Particle Counter Systems

- **Pros**

- Self contained sample delivery mechanism
- Extremely precise and small sample volume
- Variable sample volumes

- **Cons**

- Higher cost per point
- Slow sample rates
- Susceptible to bubble formation
- Not well suited to continuous sampling



Agenda



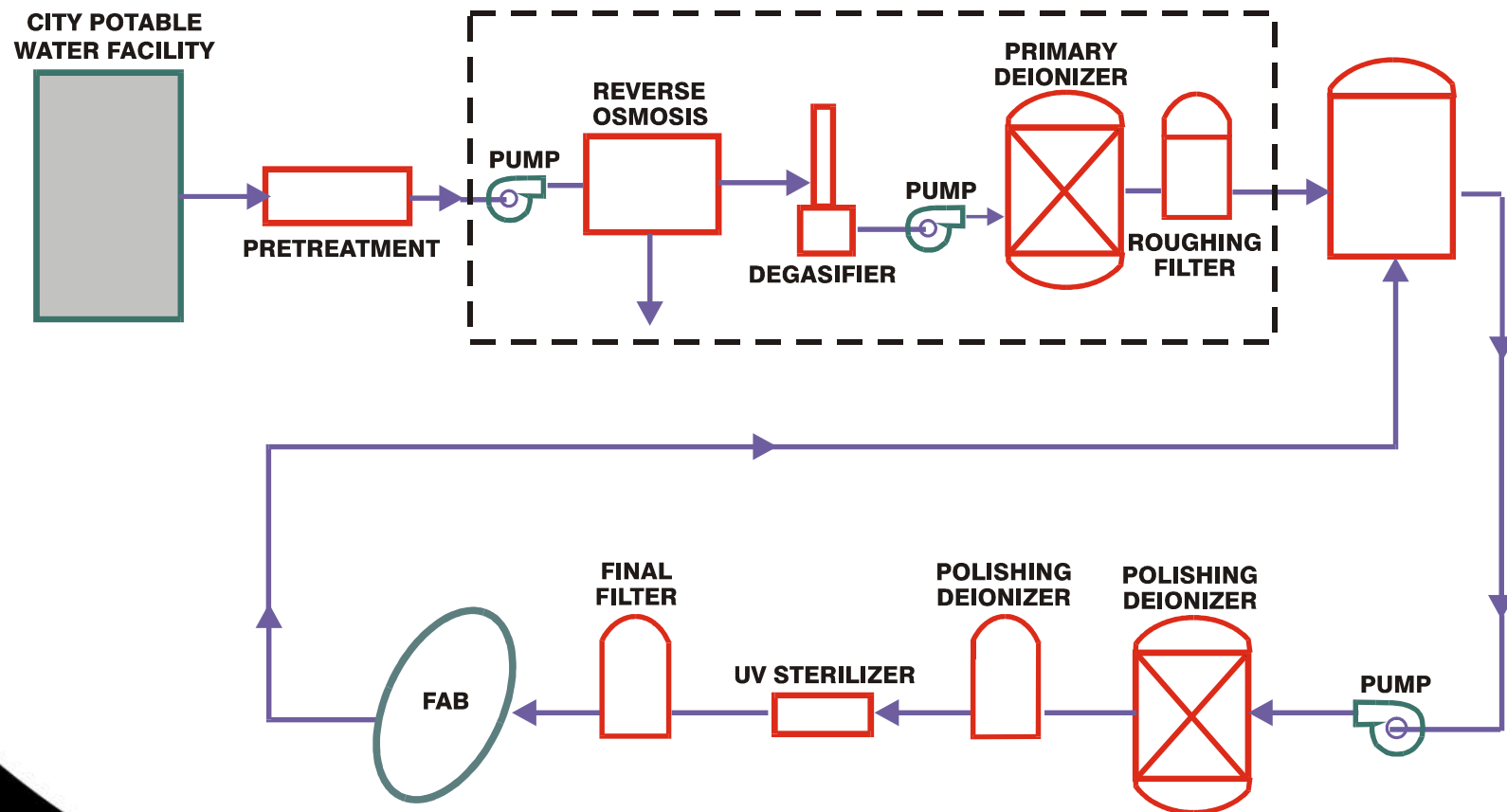
- Special considerations for liquids
- Types of liquid particle counting systems
- **DI water systems**
- Part cleanliness testing
- Chemical manufacturing and management systems
- Point-of-use wet process monitoring



DI Water Systems

- **Clean water is critical in many manufacturing systems**
 - Semiconductor
 - Disk Drive
 - Electronic and mechanical components
 - Pharmaceuticals
- **DI water is used in:**
 - Aqueous cleaning and rinsing
 - Creating chemical solutions (etching stripping, cleaning)
- **Failures in a DI water system contaminate product**
 - Particulate matter (dirt, bacteria, etc.)
 - Chemical contaminants (dissolved/colloidal silica)

DI Water Manufacturing

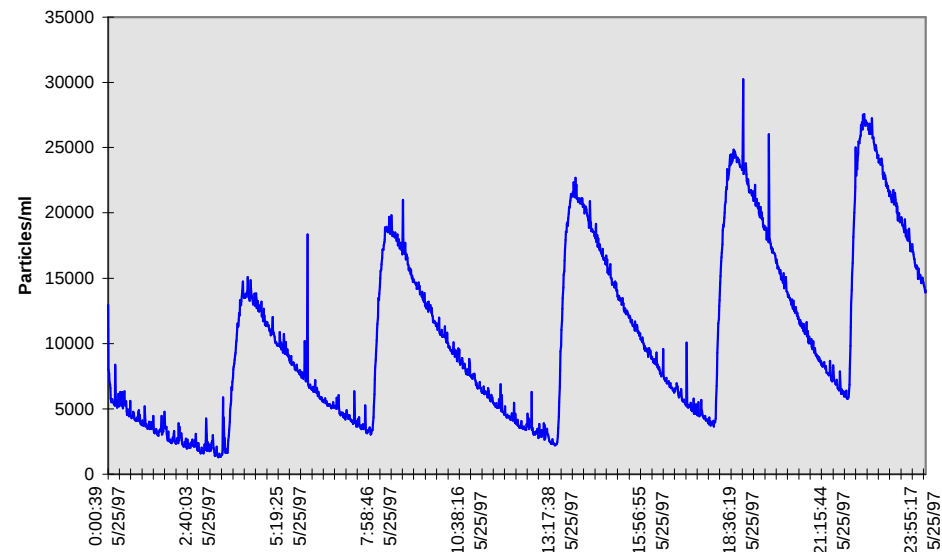


DI Water Manufacturing Pre-Treatment



- **Pre-treat output of city potable water plant.**
- **Prepares water for reverse osmosis system**
 - Failures impact life-span of RO system
- **Monitoring to 0.5 microns**
 - In-line sensor on output

Pre-Treatment Failure



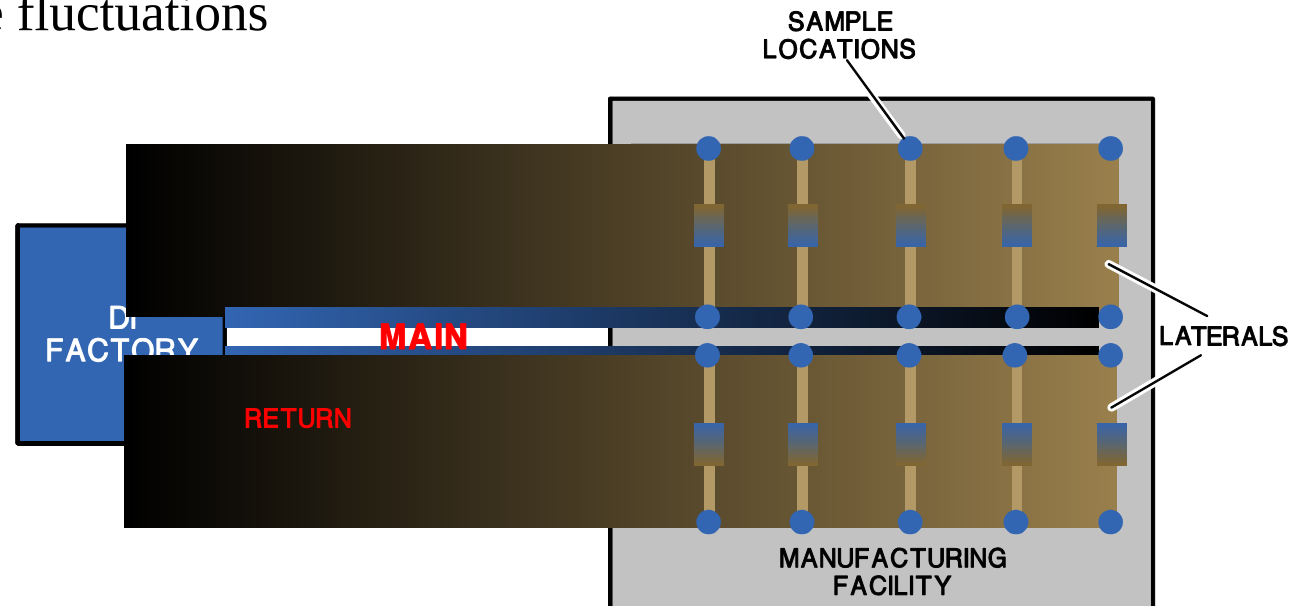
DI Water Manufacturing Final Treatment



- **Particle counters can quantify performance of:**
 - RO system (typically below degasifier)
 - Roughing filters
 - Final filter
 - DI water return
- **Most plants monitor final filters and DI water return**
 - Particulate matter
 - Silica (silica analyzer and non-volatile residue)
 - Organic carbon
 - Resistivity
 - Oxygen

DI Water Distribution

- **Deliver water from DI plant to production**
- **Return unused water to DI plant**
- **Unbalanced flow is a problem**
 - Low flow areas prone to bacteria colonies
 - Pressure fluctuations



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Chemical Manufacturing And Management Systems

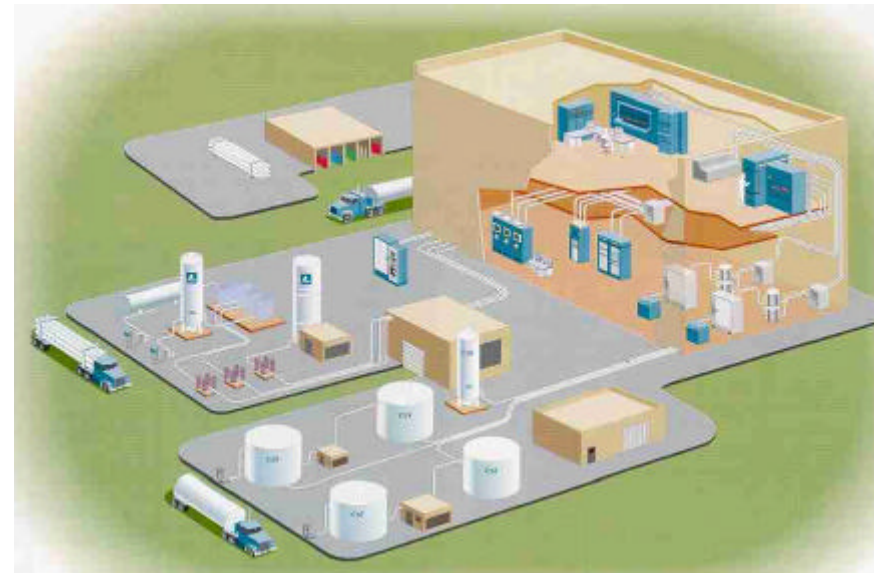


- **Particulate free chemicals**

- Required for clean manufacturing processes
- Are a competitive advantage for suppliers
- Command a higher price

- **Key monitoring points**

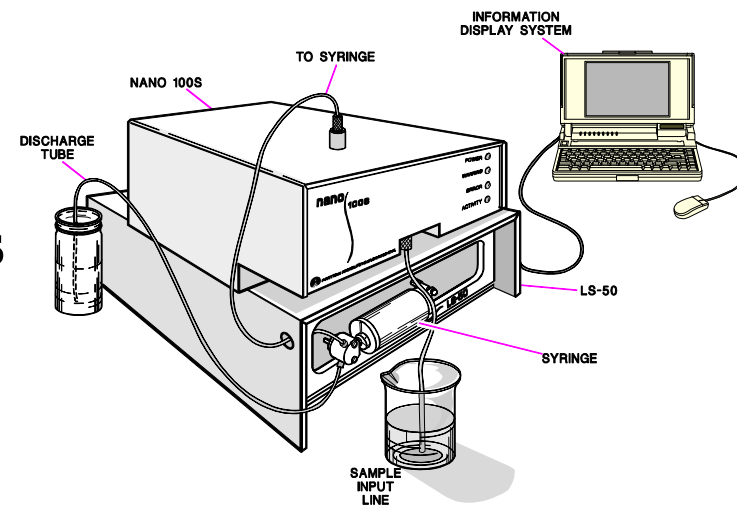
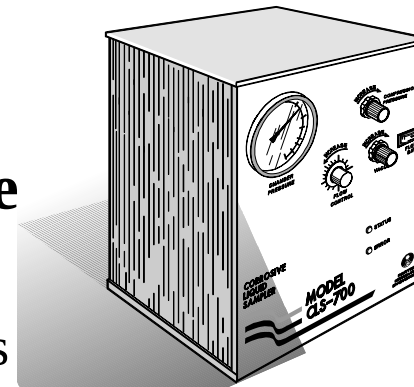
- Manufacturing / Filling system
- Distribution System
- Container assay



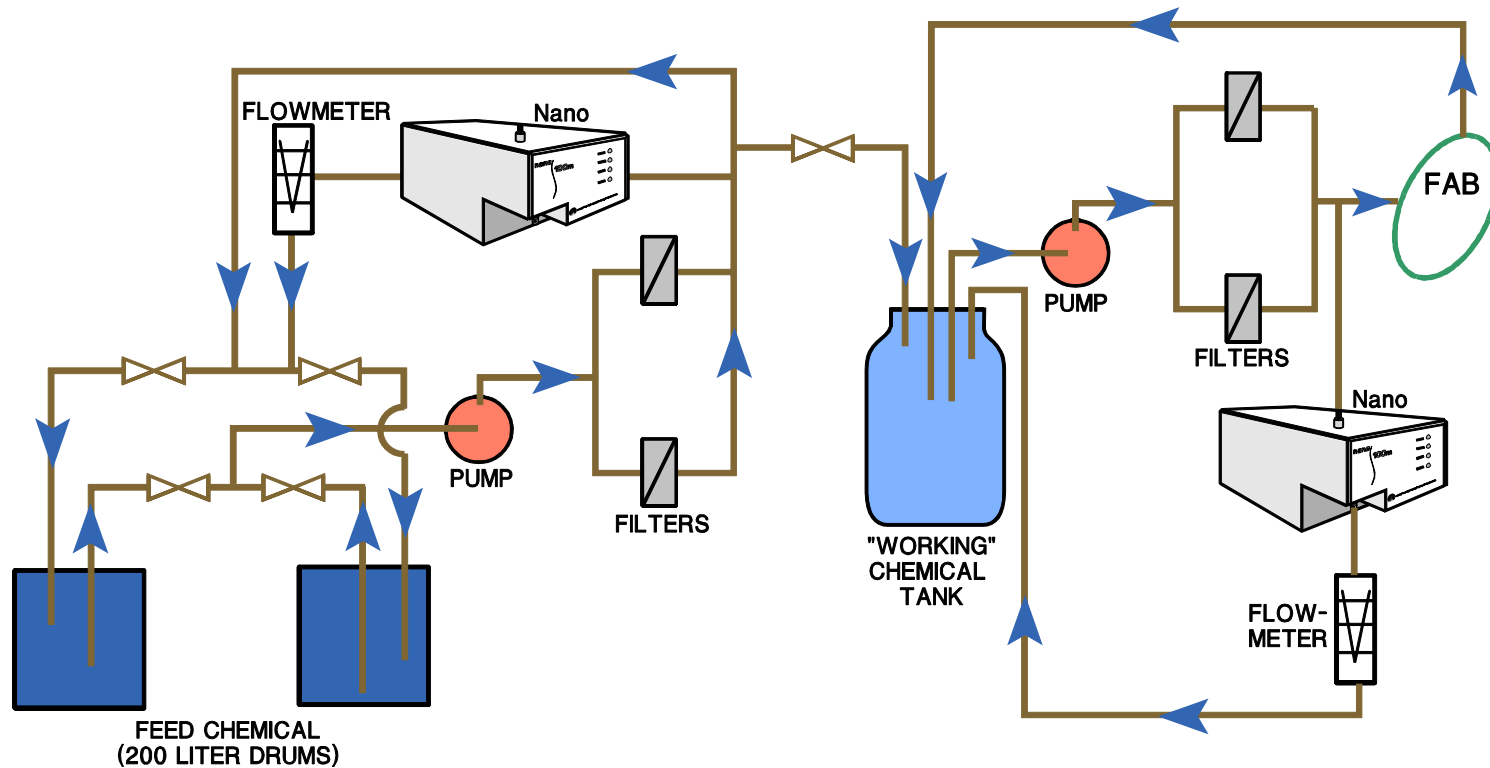
Chemical Container Assay



- Same application for suppliers and users
 - QA before shipment
 - QA before integration
- Measure contamination in a shippable container
 - Typically less stringent contamination levels
 - Batch sampling particle counters
 - Compression
 - Syringe
- QA all or representative samples



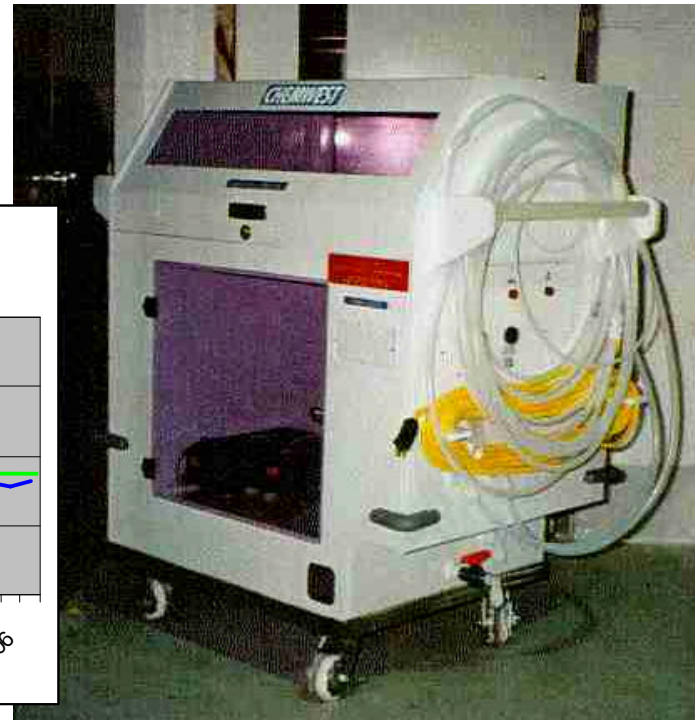
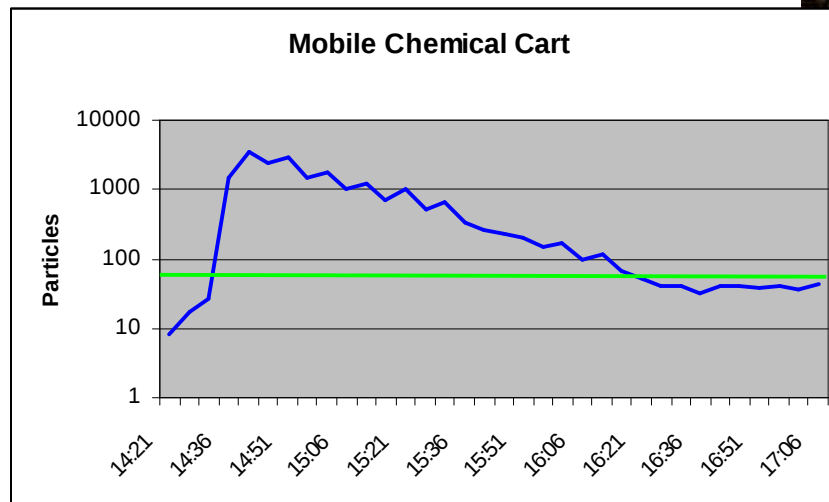
Chemical Distribution Systems



Mobile Chemical Distribution Monitoring



- **Spot-check**
 - Multiple tools
 - Distribution system
- **Allow stabilization time**



Chemical Contamination Levels



- **Contamination limits vary**
 - By industry
 - By company
 - By process
- **Limits increase downstream**
- **Typically measure 50% below size limit**

DRAM Manufacturer BCDS Limits

Chemical	Size	Limit
BOE	>0.1 μ	25 / ml
HCl	>0.1 μ	20 / ml
HF (2%)	>0.1 μ	20 / ml
HF (5%)	>0.1 μ	20 / ml
HF (49%)	>0.1 μ	25 / ml
HNO ₃	>0.1 μ	100 / ml
H ₂ O ₂	>0.2 μ	50 / ml
H ₂ SO ₄	>0.2 μ	25 / ml
H ₃ PO ₄	>0.2 μ	250 / ml
IPA	>0.1 μ	50 / ml
TMAH	>0.2 μ	500 / ml

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Chemical Process Monitoring

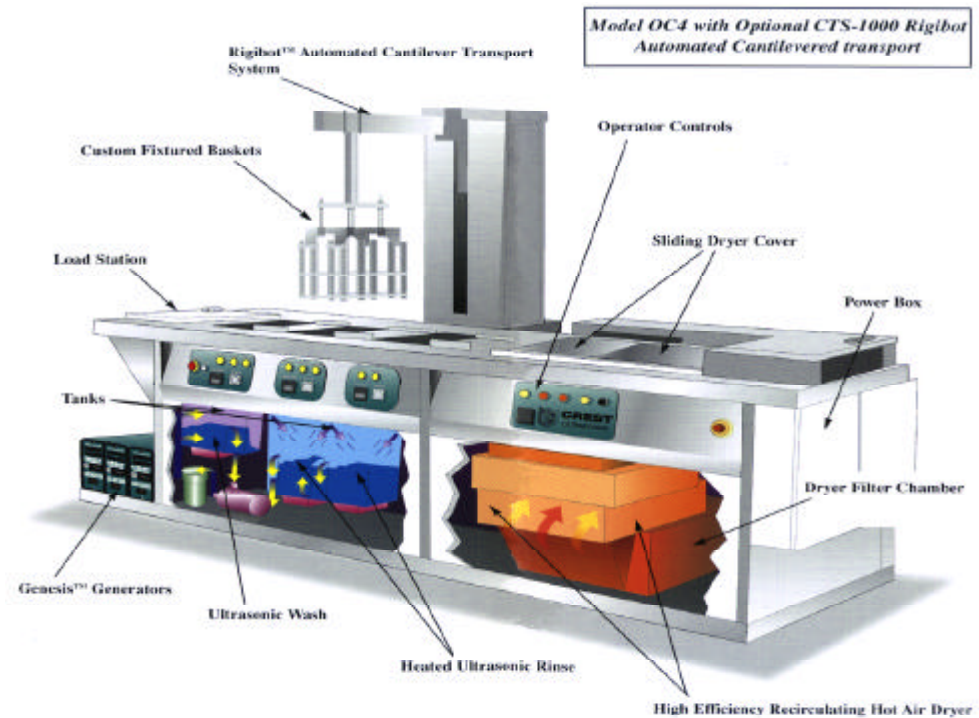


- **Monitor process cleanliness during processing**

- Cleaning
- Etching
- Coating
- Stripping
- Rinsing

- **Why**

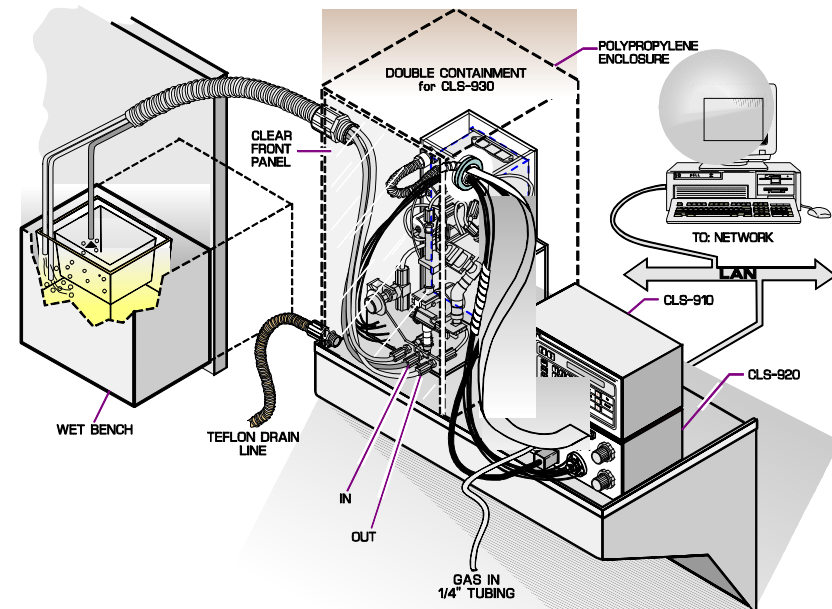
- Yield
- QA
- Process improvement



Wet Process Monitoring



- **Compression samplers**
 - Best choice for
 - Non-recirculated baths
 - Effervescent chemicals
 - Sample most process baths
 - No plumbing modification required
 - Sample fluid in contact with product
 - Sample triggered to tool events

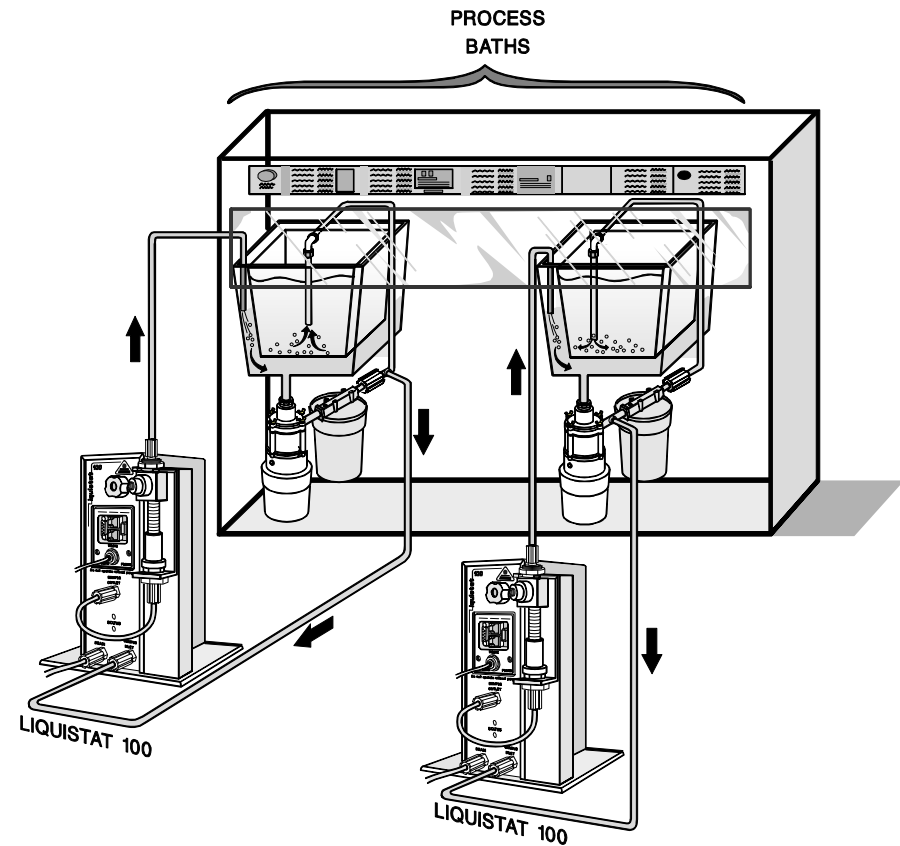


Wet Process Monitoring



■ In-line Sensors

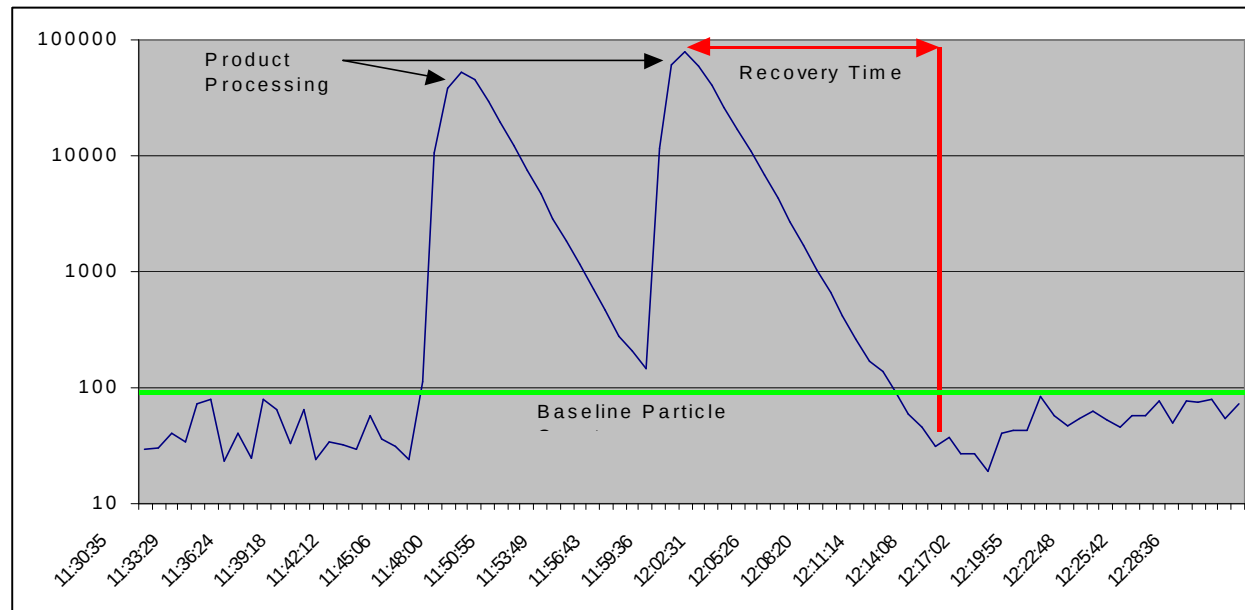
- Best choice for
 - Clean chemistries
 - Rapid processes
- Sample recirculated fluid
- Samples continuously
- Reduced costs
- Not well suited to effervescent fluids



Process Signature



- Particle surge during processing
- Cleanup to baseline depends on:
 - Filtered recirculation rate
 - Filtration efficiency
 - Continuous chemical addition



Effects of Rapid Processing

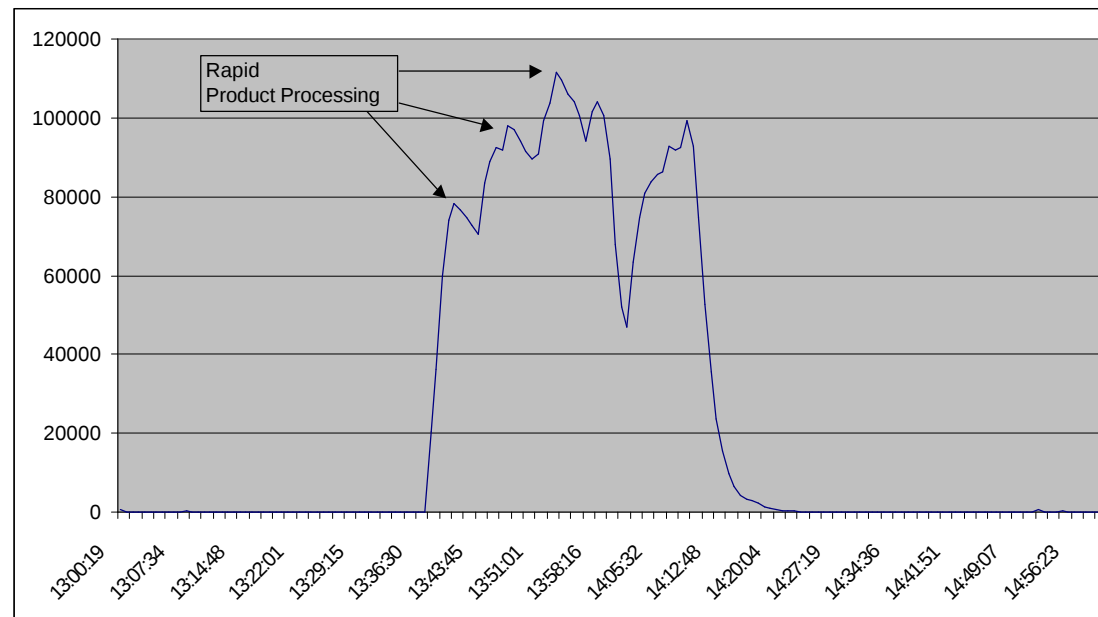


■ Rapid processing

- Incomplete return to baseline
- Increasing contamination in bath
- Inconsistent product quality

■ Solution

- Reduce processing rate
- Increase filtered re-circulation flow
- Increase fresh chemical addition

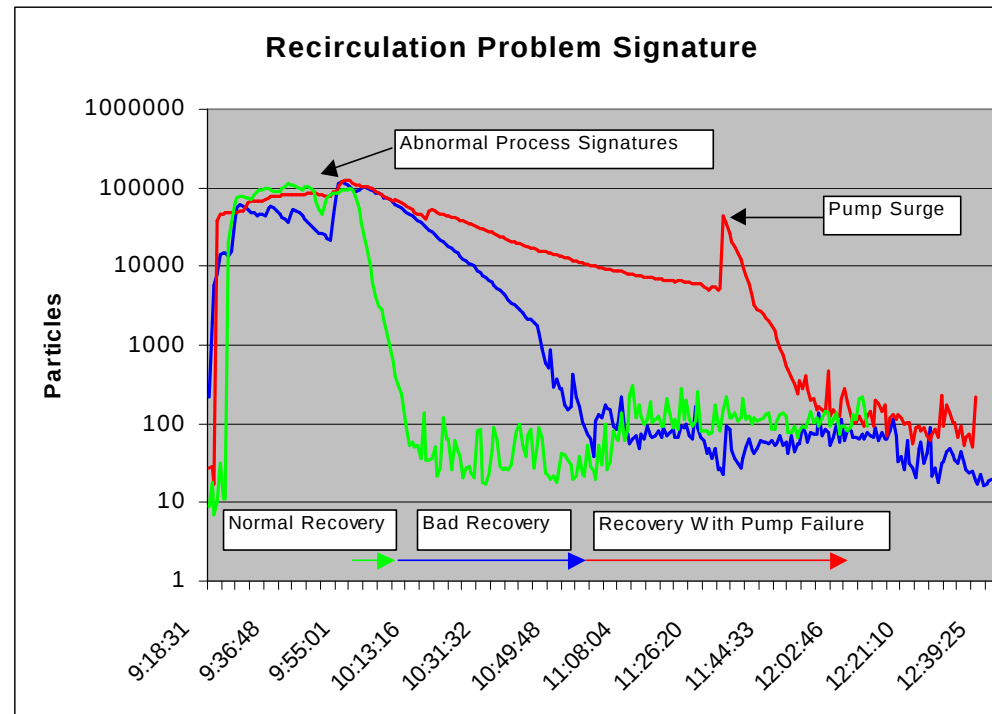


Filtration System Failures



- **Any change affecting bath cleanup can be identified**

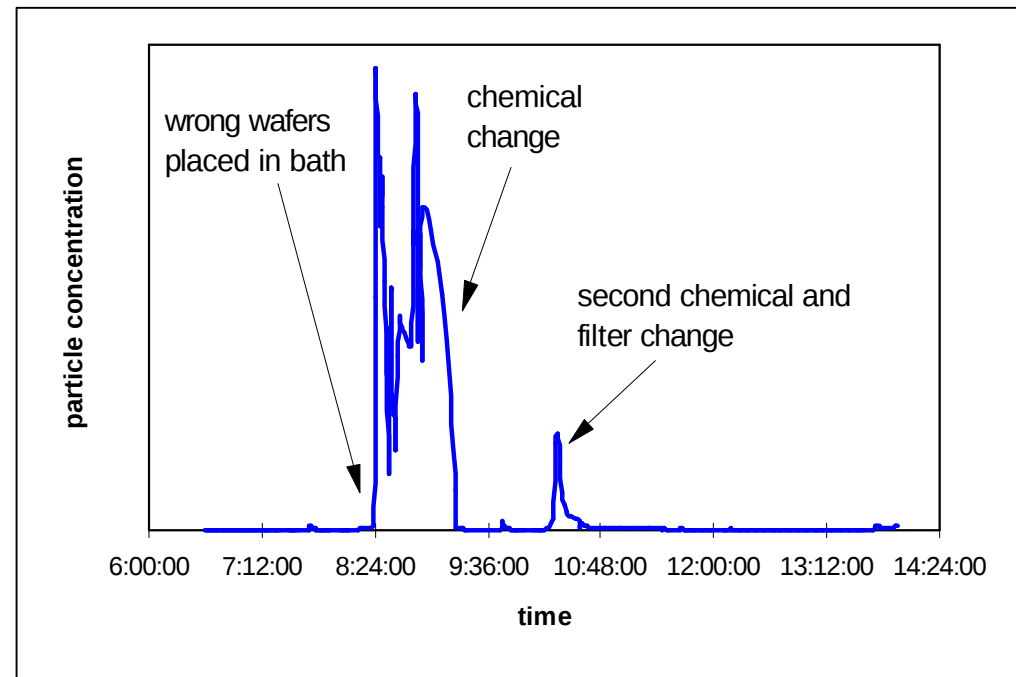
- Filter problems
 - Breakthrough
 - Poorly seated
- Pump problems
 - Failure
 - Degradation
- Plumbing problems
 - Valves
 - Fittings



Processing Failures



- Normally isolated events
- Hard to identify with surface scanning
- Types of failures
 - Incorrect sequencing
 - Upstream failure



Process Tool Qualification



- **Qualify tool for production**

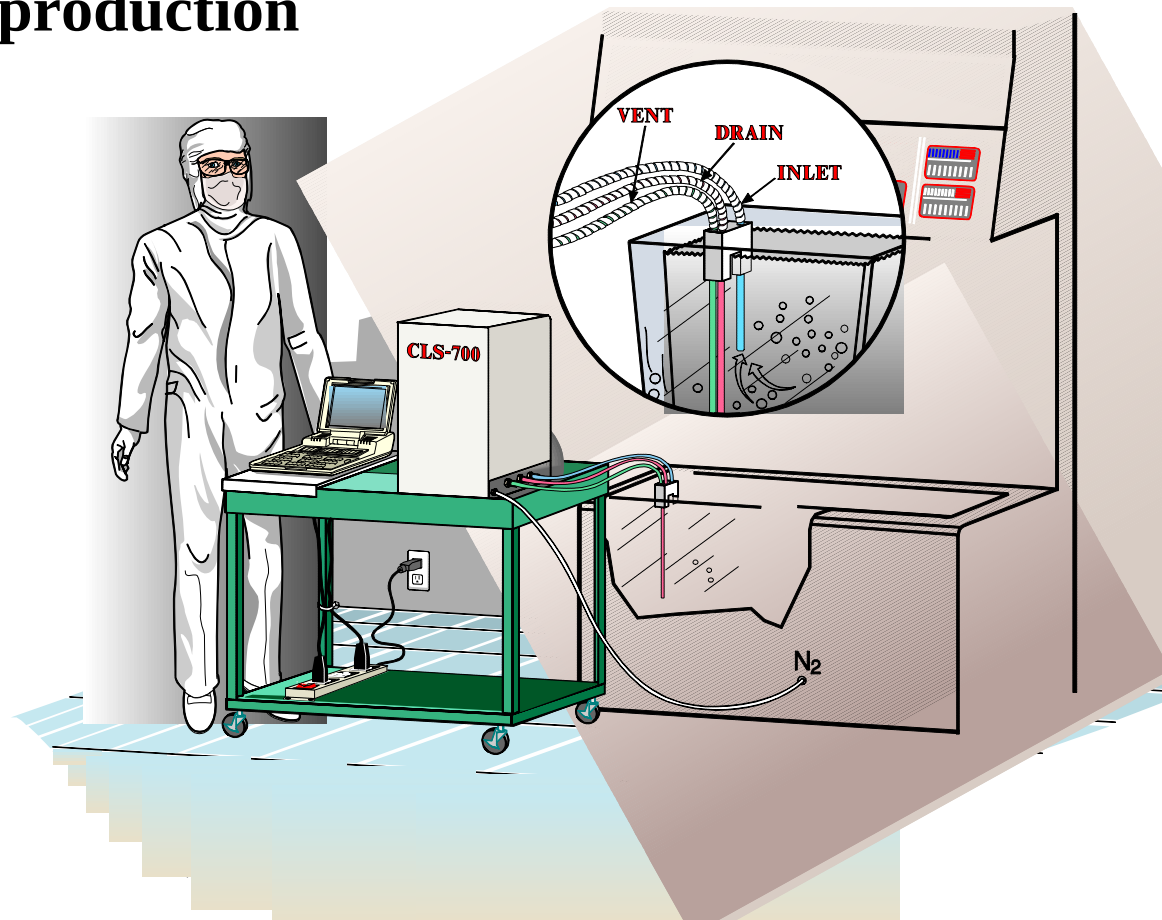
- New tool
- Post PM

- **Troubleshoot**

- Long-term
- Short-term

- **Benchmark**

- Tool A vs B
- Process Y vs Z



Summary



- **Properties of chemicals and processing systems can introduce special monitoring requirements**
 - In-line
 - Compression
 - Syringe
- **Selection of a particle counter should not be based on sensitivity alone**
- **Particle monitoring solutions are available for nearly all liquid systems**
 - Water purification
 - Chemical manufacturing
 - Chemical distribution
 - Chemical processing systems
- **Using particle counters for monitoring and controlling processes can:**
 - Improve process repeatability
 - Increase product yield