Facility Fluids Metrics and Test Methods
Technology Transfer # 95072900A-ENG
SEMATECH
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Abstract: This technology transfer was prepared by the Research Triangle Institute (RTI) as part of SEMATECH's Facility Fluids Project (S100). It is a compilation of information on existing facility fluids metric and test methods. Information on standard methods was gathered from SEMATECH and SEMI. Other information was obtained from a literature search of journals and conference proceedings. The published information primarily is concerned with the test equipment used and what levels of detection and purity were found. Many of the articles discussed the use of new equipment, either commercial or experimental. Extensive annotated bibliographies are append to the report.

Keywords: Test Methods, Standards, Purity, Test Equipment, Specifications

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1. EXECUTIVE SUMMARY

As part of the SEMATECH S100 Facility Fluids Project, the Research Triangle Institute (RTI) has compiled information on facility fluids metrics and test methods. This report serves as a reference for standards and guidelines as well as other methods that may be discussed in the literature. RTI is also performing a benchmark study of facility fluid systems cost, reliability, and purity for current fabrication facility; the benchmark study will be reported when complete.

2. INTRODUCTION

The SEMATECH S100 Facility Fluids Project has a goal of achieving lower cost gas, chemical and water distribution systems for facilities being built for 0.25 µm and 0.18 µm manufacturing processes. The specific mission was to “develop a strategic approach for design and implementation of facility fluid systems which result in low cost. These systems should be highly reliable, address purity requirements, include worker protection, minimize environmental impact, and preferably use modular components.”

Several tasks have been defined under this project. The Research Triangle Institute has been assigned two of the tasks: metrics and test methods and a performance benchmark study. Both of these tasks are to help define the current state of facility fluids systems. The S100 Project Technical Advisory Board (PTAB) has developed attributes to define the characteristics of a fluid system, especially for the systems needed in the future. These attributes focus on purity, reliability, and cost, and also include temperature, pressure, and flow. The PTAB also defined target fluids to be used for the benchmark study.

Once the attributes were defined, the Research Triangle Institute compiled existing metrics and test methods. Information on standard methods was gathered from SEMATECH and SEMI. Other information was obtained from a literature search of journals and conference proceedings. The published information primarily is concerned with the test equipment used and what levels of detection and purity were found. Many of the articles discussed the use of new equipment, either commercial or experimental. The PTAB was also asked to contribute methods, but because many of the test methods are proprietary, no methods were contributed.

This document is a compendium of the information found on metrics and test methods. It consists primarily of tables and lists of information, supplemented by extensive appendices.
3. STANDARD METHODS

3.1 SEMATECH Provisional Test Methods

SEMATECH has done extensive work on component testing, which has resulted in a series of provisional test methods. A list of these test methods can be found in Table 1, and more complete information, including abstracts can be found in Appendix A. Test methods are provided for ultrapure water system components, mass flow controllers and other gas system components. SEMATECH is working with Semiconductor Equipment and Materials International (SEMI) and the American Society for Testing and Materials (ASTM) to standardize these test methods.

Table 1. SEMATECH Provisional Test Methods for Fluids and Fluid System Components

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<thead>
<tr>
<th>Standard Number</th>
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<tr>
<td>92071220B</td>
<td>MFC</td>
<td>guide to test methods</td>
</tr>
<tr>
<td>92071221B</td>
<td>MFC</td>
<td>accuracy, linearity, repeatability, short term reproducibility, hysteresis, &amp; deadband</td>
</tr>
<tr>
<td>92071222B</td>
<td>MFC</td>
<td>reproducibility &amp; zero drift</td>
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<td>MFC</td>
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<td>92071224B</td>
<td>MFC</td>
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<tr>
<td>92071225B</td>
<td>MFC</td>
<td>verification of calibration accuracy conversion factors using surrogate gases</td>
</tr>
<tr>
<td>92071226B</td>
<td>MFC</td>
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</tr>
<tr>
<td>92071227B</td>
<td>MFC</td>
<td>determining moisture, oxygen, and total hydrocarbon contribution/retention by MFC</td>
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<tr>
<td>92071228B</td>
<td>MFC</td>
<td>determining performance characteristics from ambient and gas temperature effects</td>
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<tr>
<td>92071229B</td>
<td>MFC</td>
<td>pressure effects on indicated and actual flow</td>
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<tr>
<td>92071230B</td>
<td>MFC</td>
<td>steady-state supply voltage effects</td>
</tr>
<tr>
<td>92071231B</td>
<td>MFC</td>
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</tr>
<tr>
<td>92071232B</td>
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<td>attitude sensitivity (mounting position)</td>
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<td>92071233B</td>
<td>MFC</td>
<td>corrosion resistance</td>
</tr>
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</tr>
<tr>
<td>90120392B</td>
<td>regulators</td>
<td>performance characteristics</td>
</tr>
<tr>
<td>90120393B</td>
<td>filters</td>
<td>flow pressure drop curves</td>
</tr>
<tr>
<td>90120394B</td>
<td>valves</td>
<td>flow coefficient</td>
</tr>
<tr>
<td>90120395B</td>
<td>automatic valves</td>
<td>cycle life</td>
</tr>
<tr>
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<td></td>
<td>total hydrocarbon contribution</td>
</tr>
<tr>
<td>90120397B</td>
<td></td>
<td>moisture contribution</td>
</tr>
<tr>
<td>90120398B</td>
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<td>oxygen contribution</td>
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<tr>
<td>90120399B</td>
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<td>surface ionic/organic extractables - IC/GC/FTIR</td>
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<tr>
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<td>surface roughness by contact profilometry</td>
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<td>EDX analysis</td>
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<td>90120403B</td>
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<td>90120404B</td>
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<td>surface roughness - SEM</td>
</tr>
<tr>
<td>90160573B</td>
<td>electropolished stainless steel tubing</td>
<td>AES analysis of surface composition and chemistry</td>
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<td>93021510A</td>
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</tr>
<tr>
<td>93021511A</td>
<td>filters</td>
<td>particle contribution</td>
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<tr>
<td>92010933B</td>
<td></td>
<td>guide to test methods</td>
</tr>
<tr>
<td>92010934B</td>
<td></td>
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<td>-</td>
<td>electrical resistivity of UPW</td>
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<tr>
<td>92010936B</td>
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<td>92010937B</td>
<td>polymers</td>
<td>evaluation of bulk polymer samples</td>
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<td>92010938B</td>
<td>polymers</td>
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<td>plastic valves</td>
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<td>plastics</td>
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<tr>
<td>92010951B</td>
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<td>optical analysis of surface condition</td>
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<td>surface roughness (atomic force microscopy (AFM) method)</td>
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<td>surface roughness (scanning tunnelling microscopy method (STM))</td>
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<td>surface composition and chemical binding (electron spectrosocopy for chemical analysis (ECSA) method</td>
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Continued on the next page
Table 1. SEMATECH Provisional Test Methods for Fluids and Fluid System Components (continued)

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<td>filters</td>
<td>particle contribution</td>
</tr>
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</table>
3.2 SEMI Standards and Guidelines

The SEMI International Standards for 1994 are distributed in eleven volumes. There are separate volumes for gases and process chemicals. These volumes provide a basis for a discussion of current performance requirements, metrics and test methods. The volumes on facility standards and safety guidelines and equipment/automation and hardware provide guidelines and standard test methods for testing system components. New standards are added as they are approved and SEMI publishes a new edition of the standards each year. Contact SEMI at (415)-964-5111 for further information or to obtain specific standards.

3.2.1 SEMI Chemical Standards and Ultrapure Water Guidelines

The SEMI International Standards for process chemicals include both standards and guidelines. The base or grade 1 standards (SEMI C1) are associated with production of IC devices with critical dimension greater than 1.2 μm. Grade 2 (SEMI C7) has tighter specifications, generally on the order of 10 ppb for trace metals. Grade 2 is associated with dimensions between 0.8 and 1.2 μm. There are also guidelines for future production needs. Tier A guidelines are labeled SEMI C7, correspond to (and become) Grade 2 standards upon approval. Tier B (SEMI C8) have tighter specifications, are recommendations for dimensions between 0.2 and 0.6 μm, and will become Grade 3 standards when approved. Tier C guidelines (SEMI C12) will describe specifications needed for 0.09 to 0.2 μm dimensions. The Tier guidelines are developed by the North American Process Chemicals Committee. The European Gases and Chemicals Committee have developed the VLSI grade guidelines for dimensions of 0.8 to 1.2 μm (SEMI C11).

Section 3 of the SEMI C1-94, Specification for reagents, gives general procedures and guidelines for certain analytical methods, including
1. wide bore column gas chromatography,
2. color,
3. determination of residue after evaporation,
4. reagents,
5. determination of trace elements by emission spectrography,
6. determination of trace elements by atomic absorption spectrography,
7. determination of trace elements by flame emission spectrography,
8. use of a pH meter,
9. particle measurement,
10. determination of trace elements by plasma emission spectrography,
11. determination of various ions by ion chromatography,
12. determination of trace elements by graphite furnace atomic absorption spectrography, and
13. determination of trace elements by inductively coupled plasma mass spectrography.

Individual standards also give specific, detailed information on necessary preparation and analysis. For the trace metals, Grade 1 (base level) specifications specify flame atomic absorption spectroscopy for Group 1 elements and plasma emission spectroscopy for the rest. For Grade 2, GFAA is used for sodium, potassium, and iron, while ICP/MS is used for the rest. Optical particle counters are used for particle measurement.
A listing of the 1994 chemical standards are given in Table 2. Specific maximum allowable contaminant levels related to the SI00 benchmark study are shown in Table 3. Table 4 gives information on the SEMI ultrapure water guidelines which are applicable to the benchmark study.

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<th>Standard Grade</th>
<th>Guideline Tier</th>
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<td>electronics</td>
<td>SEMI C1.24-91</td>
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<tr>
<td>1, 1, 1 Trichloroethane (provisional)</td>
<td>furnace</td>
<td>SEMI C1.26-92</td>
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<tr>
<td>1, 1, 1 Trichloroethane (furnace grade)</td>
<td>A</td>
<td>SEMI C7.15-91</td>
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<tr>
<td>2-Propanol</td>
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<td></td>
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<tr>
<td>2-Propanol</td>
<td>2</td>
<td>SEMI C7.7-93</td>
<td></td>
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<tr>
<td>2-Propanol</td>
<td>B</td>
<td>SEMI C8.7-92</td>
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<tr>
<td>Acetic Acid</td>
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<td>SEMI C1.1-91</td>
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<td>1</td>
<td>SEMI C1.2-91</td>
<td></td>
</tr>
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<td>Ammonium Fluoride solution</td>
<td>VLSI</td>
<td>SEMI C11.2-94</td>
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<td>VLSI</td>
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<td>Dichlorugene (methylene chloride)</td>
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<td>Hydrofluoric Acid (10:1 v/v) (4.9%)</td>
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Continued on next page
Table 2. SEMI International Standards and Guidelines for Process Chemicals, 1994 (Continued)

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SEMI suggested guidelines for pure water for semiconductor processing.
Table 3. SEMI International Standards and Guidelines for Chemicals, 1994
Maximum Acceptable Levels, Relative to SEMATECH S100 Benchmark Study

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Level indicates standards (Grade 1 or 2) and guidelines (VLSI and A, B Tiers)
Units - Metals, Grade 1, VLSI Grade, ppm; Grade 2, Tier A, Tier B, ppb
Particles (#/ml) - greater than 1 µm, Grade 1; greater than 0.5 µm, rest
Table 4. SEMI Suggested Guidelines for Pure Water, 1994
Semiconductor Processing, Maximum Acceptable Levels
Relative to SEMATECH S100 Benchmark Study

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<th>Impurity</th>
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<td>Boron - ppb</td>
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<td>TOC - ppb</td>
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<tr>
<td>Ions - ppb</td>
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<tr>
<td>Cl-</td>
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<tr>
<td>Br-</td>
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<td>-</td>
</tr>
<tr>
<td>NO3-</td>
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<td>Particles (by SEM) - #/L</td>
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<td>Particles (by on-line laser) - #/L</td>
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<td>&gt; 0.5 U</td>
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SEMATECH  

Technology Transfer #95072900A-ENG
Table 5. Current Activity on SEMI Standards and Guidelines for Process Chemicals (Continued)

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<th>Standard Number</th>
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<td>Three year review: guidelines for n-methyl-2-pyrrolidone, tier A</td>
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<tr>
<td>SEMI C12.1</td>
<td>Guideline for ammonium hydroxide, tier C (new)</td>
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<td>SEMI C12.2</td>
<td>Guideline for hydrochloric acid, tier C (new)</td>
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<td>SEMI C12.3</td>
<td>Guideline for 4.9% hydrofluoric acid, tier C (new)</td>
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<td>SEMI C12.4</td>
<td>Guideline for hydrogen peroxide, tier C (new)</td>
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<td>3-year reviews of</td>
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<td>Standard for acetic acid</td>
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<td>Standard for acetone</td>
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<td>Standard for n-butyl acetate</td>
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<td>Standard for dichloromethane</td>
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<td>Standard for methanol</td>
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<td>SEMI C1.15-91</td>
<td>Standard for 2-propanol</td>
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<td>Standard for 80% phosphoric acid</td>
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<td>Standard for 85% phosphoric acid</td>
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<td>Standard for 4.9% hydrofluoric acid (10:1 v/v), grade 2</td>
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<td>Guideline for hydrofluoric acid, VLSI grade</td>
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<td>Guideline for 2-propanol, VLSI grade</td>
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<td>Addition to SEMI C1, Specifications for Reagents</td>
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3.2.2 SEMI Standards for Gases

The specifications for gases SEMI 3-94 is incomplete. Gas chromatography will be added later. Information on special analyses include

1. resistivity measurement,
2. metal analysis of gaseous silicon compounds, including Fourier transform infrared spectroscopy (FTIR), photoluminescence spectroscopy (PLS), atomic absorption spectrophotometry (AA), inductively coupled plasma emission spectrometry (ICP), and ion chromatography (IC).

A listing of the 1994 chemical standards are given in Table 6. Specific maximum allowable contaminant levels related to the S100 benchmark study are shown in Table 7.
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All units ppm except where specified otherwise

Particles to be determined between supplier and user

THC exceptions - (a - C1 - C4), (b - other halogenated hydrocarbons), (c - methane), (d - C1 - C5)

CO, CO2 - value shown in the middle of the two columns indicate that value is sum of both contaminants

w/Ar, w/O2, w/N2 - indicates that value is sum of multiple contaminants
SEMI is currently working on standards and guidelines as shown in Table 8.

### Table 8. Current Activity on SEMI Standards and Guidelines for Gases

<table>
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<th>Standard Number</th>
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<td>Standard for silane (SiH₄)</td>
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<tr>
<td>SEMI C3.47-93</td>
<td>Standard for hydrogen bromide (HBR)</td>
</tr>
<tr>
<td>SEMI C3.50</td>
<td>Standard for nitrous oxide (N₂O) (new)</td>
</tr>
<tr>
<td>SEMI C3.51</td>
<td>Standard for boron trichloride, 99.98% quality (new)</td>
</tr>
<tr>
<td>SEMI C3.52</td>
<td>Standard for tungsten hexafluoride, 99.996% quality (new)</td>
</tr>
<tr>
<td>SEMI C13</td>
<td>Particle test methods for point-of-use filters (new)</td>
</tr>
<tr>
<td>SEMI C14</td>
<td>Particle test methods for 10-inch gas filter cartridges (new)</td>
</tr>
<tr>
<td>SEMI C15</td>
<td>Test methods for PPM and PPB humidity standards (new)</td>
</tr>
</tbody>
</table>

#### 3-year reviews of

- SEMI C3.21-90 Standard for carbon tetrafluoride in cylinders
- SEMI C3.29-90 Standard for nitrogen, VLSI grade bulk gaseous
- SEMI C3.30-90 Standard for hydrogen, VLSI grade bulk
- SEMI C3.38-89 Standard for tungsten hexafluoride, 99.996% quality
- SEMI C3.43-90 Standard for hydrogen fluoride, anhydrous

#### and additional work on

- SEMI C3.10-92 Standard for silane
- SEMI C3.21-90 Standard for carbon tetrafluoride in cylinders
- SEMI C3.41 Standard for oxygen, VLSI grade bulk
- SEMI C3.42 Standard for argon VLSI grade bulk
- SEMI C3.51-95 Standard for boron trichloride, 99.98% quality
- SEMI C3.52-95 Standard for tungsten hexafluoride, 99.996% quality new standard for trifluoromethane (CHF₃).
3.2.3 SEMI Facility and Component Standards and Safety Guidelines

SEMI provides standards and guidelines for components, facility handling, and safety. A listing of standards related to fluid systems is shown in Table 9.

<table>
<thead>
<tr>
<th>Standard Number</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>SEMI E12-91</td>
<td>Standard for standard pressure and standard temperature for flow units used in mass flow meters and mass flow controllers</td>
</tr>
<tr>
<td>SEMI E16-90</td>
<td>Guideline for determining and describing mass flow controller leak rates</td>
</tr>
<tr>
<td>SEMI E17-91</td>
<td>Guideline for mass flow controller transient characteristics tests</td>
</tr>
<tr>
<td>SEMI E18-91</td>
<td>Guideline for temperature specifications of the mass flow controller</td>
</tr>
<tr>
<td>SEMI E27-92</td>
<td>Standard for mass flow controller and mass flow meter linearity</td>
</tr>
<tr>
<td>SEMI E28-92</td>
<td>Guideline for pressure specifications of the mass flow controller</td>
</tr>
<tr>
<td>SEMI E29-93</td>
<td>Standard terminology for the calibration of mass flow controllers and mass flow meters</td>
</tr>
<tr>
<td>SEMI F1-90</td>
<td>Specification for leak integrity of toxic gas piping systems</td>
</tr>
<tr>
<td>SEMI F2-94</td>
<td>Specification for 316L stainless steel tubing for general purpose semiconductor manufacturing</td>
</tr>
<tr>
<td>SEMI F3-94</td>
<td>Guide for welding stainless steel tubing for semiconductor manufacturing applications</td>
</tr>
<tr>
<td>SEMI F4-90</td>
<td>Guide for remotely actuated cylinder valves</td>
</tr>
<tr>
<td>SEMI F5-90</td>
<td>Guide for gaseous effluent handling</td>
</tr>
<tr>
<td>SEMI F6-92</td>
<td>Guide for secondary containment of hazardous gas piping systems</td>
</tr>
<tr>
<td>SEMI F7-92</td>
<td>Test method to determine the tensile strength of tube fitting connections made of fluorocarbon materials</td>
</tr>
<tr>
<td>SEMI F8-92</td>
<td>Test method for evaluating the sealing capabilities of tube fitting connections made of fluorocarbon materials, when subjected to tensile forces</td>
</tr>
<tr>
<td>SEMI F9-92</td>
<td>Test methods to determine the leakage characteristics of tube fitting connections made of fluorocarbon materials when subjected to a side load condition</td>
</tr>
<tr>
<td>SEMI F10-93</td>
<td>Test method to determine the internal pressure required to produce a failure of a tube fitting connection made of fluorocarbon materials</td>
</tr>
<tr>
<td>SEMI F11-93</td>
<td>Test method to obtain an indication of the thermal characteristics of tube fitting connections made of fluorocarbon materials</td>
</tr>
<tr>
<td>SEMI F12-93</td>
<td>Test method to determine the sealing capabilities of fittings, made of fluorocarbon material, after being subjected to a heat cycle</td>
</tr>
<tr>
<td>SEMI F13-93</td>
<td>Guide for gas source control equipment</td>
</tr>
<tr>
<td>SEMI F14-93</td>
<td>Guide for the design of gas source equipment enclosures</td>
</tr>
<tr>
<td>SEMI F15-93</td>
<td>Test method for enclosures using sulfur hexafluoride tracer gas and gas chromatography</td>
</tr>
</tbody>
</table>

Continued on next page
Table 9. SEMI Fluid Facility and Component Standards, 1994 (Continued)

<table>
<thead>
<tr>
<th>Standard Number</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>SEMI F16-94</td>
<td>Specification for 3 16L stainless steel tubing which is to be finished and electropolished for high purity</td>
</tr>
<tr>
<td>SEMI F17-90</td>
<td>Safety guideline for visual hazard alerts</td>
</tr>
<tr>
<td>SEMI F18-93</td>
<td>Safety guidelines for semiconductor manufacturing equipment</td>
</tr>
<tr>
<td>SEMI S3-91</td>
<td>Safety guidelines for heated chemical baths</td>
</tr>
<tr>
<td>SEMI S4-92</td>
<td>Safety guideline for the segregation/separation of gas cylinders contained in cabinets</td>
</tr>
<tr>
<td>SEMI S5-93</td>
<td>Safety guideline for flow limiting devices</td>
</tr>
<tr>
<td>SEMI S6-93</td>
<td>Safety guideline for ventilation</td>
</tr>
<tr>
<td>SEMI S7-94</td>
<td>Safety guideline for third party environmental, health, and safety equipment evaluation</td>
</tr>
</tbody>
</table>

SEMI is currently working with SEMATECH to standardize test methods developed by SEMATECH. Work in progress (April 1995) includes the following:

SEMI draft document #2335, based on information in SEMASPEC 90120400B-STD, test method for determination of surface roughness by contact profilometry for gas distribution components.
SEMI draft document #2336, based on information in SEMASPEC 90120402B-STD, test method for EDX analysis of metallic surface condition for gas distribution system components.
SEMI draft document #2337, based on information in SEMASPEC 90120403B-STD, test method for ESCA analysis of surface composition and chemistry of electropolished stainless steel tubing for gas distribution system components.

In addition, the SEMI Mass Flow Controllers Task Force is reviewing the following SEMASPEC test methods:

92071221B-STD SEMASPEC provisional test method for determining accuracy, linearity, repeatability, short term reproducibility, hysteresis, and deadband of thermal mass flow controllers.
92071222B-STD SEMASPEC provisional test method for determining reproducibility and zero drift for thermal mass flow controllers.
92071224B-STD SEMASPEC provisional test method for determining reliability of thermal mass flow controllers.
92071225B-STD SEMASPEC provisional test method for verification of calibration accuracy and calculation of conversion factors for a mass flow controller using surrogate gases thermal mass flow controllers.
92071226B-STD SEMASPEC provisional test method for determining particle contribution by thermal mass flow controllers.
92071228B-STD SEMASPEC provisional test method for determining mass flow controller performance characteristics from ambient and gas temperature effects.
92071229B-STD SEMASPEC provisional test method for determining pressure effects on indicated and actual flow for thermal mass flow controllers.
3.3. ASTM Semiconductor-Related Standards

ASTM has made standards of some of the SEMASPEC and other semiconductor-related test methods as shown in Table 10. Some editorial changes have been made in individual standards.

<table>
<thead>
<tr>
<th>Method Number</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1094-87 (1992)</td>
<td>Test methods for microbiological monitoring of water used for processing electron and microelectronic devices by direct-pressure tap sampling valve and by the preserilized plastic bag method</td>
</tr>
<tr>
<td>F1095-88 (1994)</td>
<td>Test method for rapid enumeration of bacteria in electronics-grade purified water systems by direct-count epifluorescence microscopy</td>
</tr>
<tr>
<td>F1228-89 (1994)</td>
<td>Test method for oxidizable (organic) carbon on wafer surfaces (by persulfate)</td>
</tr>
<tr>
<td>F1372-93</td>
<td>Test method for scanning electron microscope (SEM) analysis of metallic surface condition for gas distribution system components</td>
</tr>
<tr>
<td>F1373-93</td>
<td>Test method for determination of cycle life of automatic valves for gas distribution system components</td>
</tr>
<tr>
<td>F1374-92</td>
<td>Test method for determination of cycle life of automatic valves for gas distribution system components</td>
</tr>
<tr>
<td>F1375-92</td>
<td>Test method for energy dispersive x-ray spectrometer (EDX) analysis of metallic surface condition for gas distribution system components</td>
</tr>
<tr>
<td>F1376-92</td>
<td>Guide for metallurgical analysis for gas distribution system components</td>
</tr>
<tr>
<td>F1394-92</td>
<td>Test method for determination of particle contribution from gas distribution system valves</td>
</tr>
<tr>
<td>F1396-93</td>
<td>Test method for determination of oxygen contribution by gas distribution system components</td>
</tr>
<tr>
<td>F1397-93</td>
<td>Test method for determination of moisture contribution by gas distribution system components</td>
</tr>
<tr>
<td>F1398-93</td>
<td>Test method for determination of total hydrocarbon contribution by gas distribution system components</td>
</tr>
<tr>
<td>F1438-93</td>
<td>Test method for determination of surface roughness by scanning tunneling microscopy for gas distribution system components.</td>
</tr>
</tbody>
</table>
4. OTHER METHODS

The semiconductor industry is continually improving purity of facility fluids. The test equipment manufacturers, therefore continually improve their products to provide better levels of detection. While it would be ideal to have a list of test methods and/or equipment and the limits of each, this is not practical. In practice, it is often possible to combine various techniques to improve the level of detection. Many of these techniques are not published, rather they are confidential within a company, provided by equipment suppliers, or passed on by word of mouth. One such example is using an inductively coupled plasma mass spectrometer (ICP/MS) to detect trace levels of metals. SEMI C1-94, Specifications for reagents, paragraph 3.13 gives guidelines for the determination of trace elements by ICP/MS. This method is used for grade 2, with measurements in the ppb range. Two separate techniques can be used in conjunction with the ICP/MS, either separately or in tandem, to increase the sensitivity of the analysis. First, a chelation concentration technique can be used so that the sample flows through a chelation resin column which selectively retains transition metals. If the resulting sample is then transferred directly to the nebulizer of the ICP, there can be a detection limit of at least 100-fold. A commercial chelation system is available. The second technique is to use a high efficiency nebulizer. Multiple commercial versions are available which involved redesigned spray chambers or special nebulization hardware. These nebulizers may increase the sensitivity 10-fold in some cases. Using the two techniques together, it is possible to increase sensitivity 1000-fold, thus allowing for low ppt measurements.

The scope of this project did not allow for collection of facility fluids test methods currently in use by the SEMATECH members. Instead, a literature search was conducted. The primary focus was on testing for purity within the fluid distribution systems, although the bibliography also includes some articles on cost, reliability, system component testing, purity testing at the point of use and tool, and information on generation, reclaim, and recycling of the fluids. Some of the articles discuss the use of new commercial or experimental equipment, others only mention the equipment used and focus on the measurements taken or the facility fluid system itself. Only in rare cases are there details given about the test procedures; it is much more common to mention just the test equipment used. The abstracts given in Appendix B have in cases been supplemented by information on the test equipment used.
4.1 Subject Cross Reference Listing for Bibliography

To facilitate the use of the bibliography, there is a cross reference below for testing and analysis of process chemicals, gases, and water. The references are listed by first author and year and refer to the annotated bibliography in Appendix B. If specific methods or equipment are given in the article, they are listed with the reference.

Chemicals
General References
Camenzind 1991, trace organics
Cawthon 1992, ICP-MS, GFAA
Grant, Smith et. al. 1994, ICP-MS, GFAAS, particle counter
Hutton 1993
Ikeda 1992
Ishihara 1992
Isikikawa 1992
Kastle 1991
O’Dougherty 1994
Sugawara 1990, GFAA, ACP-MS, liquid ion chromatograph
Vargason 1990
Wang 1994
Wear 1994

References for Particle Measurement
Blackford 1992, interferometer
Capitanio 1993, particle monitors
Cooper 1989, review
Donovan 1990, book
Dab 1990
Grant 1989
Grant & Heilser 1993
Grant 1994
Gruber 1990
Hurd 1992
Hurd, Crocker, et.al. 1992
Knollenberg 1991
Krygier 1985
Lieberman 1990
Singer 1992
Willis 1989
Zorn 1987

References for Measurement of Metals (Na, K, Fe, Cu, Al, Zn, Cr, Ni)
Fitzgerald 1992, ICP-MS, GFAA, ICP-AE
Grant, Van Dyke, Wilkes 1993, ICP-AES, GFAAS
Gupta 1992
Rath 1990
Tan 1992
Gases

General References
Bhadha 1994, inhouse methods, also APIMS
Ezell 1993, APIMS, RGA, particle analyzers
Goddard 1994, APIMS
Goddard 1991, APIMS, custom monitors
Ketkar & Bzik 1993, APIMS
Ketkar 1992, APIMS
Ketkar, Scott, et al. 1994, APIMS
Leggett 1992, APIMS
Leggett 1993, APIMS
Lowles 1986
Ma 1992, APIMS
Maroulis 1992, APIMS
McAndrew 1993
Mulready 1992
Ohki 1994, leaks, APIMS
Plante 1991
Plante 1994
Ridgeway, Ketkar, Zatko, et al. 1992, LODs
Rosamia 1994, ICP-HRMS
Sanborn 1993
Schmitt 1993, CNC, moisture, FTIR
Siefering 1992 (2 articles)
Siefering 1994
Tabler 1993
Wang 1992 (2 articles)
Wear 1992

References for Measurement of Molecular Contamination, O₂, THC, CO, CO₂, H₂, N₂
Ma 1994, APIMS
Ostrander 1992, RGA
Ridgeway, Ketkar, Martinez de Pinillos 1992, THC APIMS
Whitlock 1988

References for Measurement of Moisture
Athalaye 1994, APIMS
Briesacher 1992, moisture generator to calibrate APIMS
Brzuchcy 1994, FTIR for chlorine
Buck 1993, chilled mirror hygrometer
Ezell 1992, APIMS, hygrometers
Ezell 1994, FTIR
McAndrew 1991 (2 articles)
McAndrew 1992
McDemott 1991
Nakamura 1992 APIMS
Pfeifer 1994, various sensors
Rowe 1994, FTIR, corrosive gases
References for Measurement of Moisture (continued)
    Siefering 1993
    Snow 1994, on line analysis
    Succi 1994
    Tom 1993, in line monitor
    Yesenofski 1994, oscillating crystal hygrometer

References for Measurement of Particles
    Borkman 1991
    Borkman, Couch, Malczewski 1992
    Cooper 1989, review
    Davison 1990, review
    Donovan 1990, book
    Gerristead 1990, review
    Holmer 1993, CNC
    Kibbs 1992
    Liu 1989
    Sommer 1990, optical
    Sommer 1992, CNC
    Thorogood 1989
    Wang 1993, new sensor

Water
General References
    Carmody 1990
    Daily 1993
    Firth 1991a
    Governa 1991
    Homnick 1992
    Homnick 1993
    Husted 1994
    Martyak, Carmody, Lindahl 1992, (2 articles)

References for Measurement of Particles
    Anderson 1990
    Balazs 1988
    Balazs 1989, SEM, TEM, AEM, ESCA, FTIR, Auger, Xray
    Grant 1993, interferometer
    Kearney 1989
    Knollenberg 1992
    Livingston 1990
    Martyak, Carmody, Clancy 1991
    Meltzer 1993 (book)
    Van Sickle 1993
    Yang 1989
References for Measurement of Molecular Contamination, Silica, TOC, TDS, dissolved Oxygen, Boron, other organics, process specific reactants, collection system contaminants, post CMP

Anderson 1986, TOC analyzer
Blackford 1991, nonvolatile residue
Firth 1991b, TOC
Hutte 1993, TOC analyzer
Kawada 1994
Pate 1991
Poirier 1985
Yagi 1994

References for Measurement of Bacteria
Carpenter 1991 (2 references)
Gould 1990
Martyak 1992
Martyak 1993
Meltzer 1993 book
Pepper 1994
Yagi 1992

References for Measurement of pH
Gray 1989

References for Measurement of Conductivity and Resistivity
Gray 1988

General

References for Measurement of pH
Galster, 1991 (book)
Wescott 1978 (book)

References for Measurement of Temperature, Pressure, Flow for All Fluids
Benedict 1984 (book), temperature, pressure, and air flow
DeCarlo 1984 (book), flow
Kerlin 1982 (book), temperature
Michalski 1991 (book), temperature
Miller 1989 (book), flow
Pavese 1992 (book), temperature and pressure
Quinn 1990 (book), temperature
Spitzer 1990 (book), flow

5. SUMMARY

As part of the SEMATECH S100 Facility Fluids Project, the Research Triangle Institute has provided information on current standard metrics test methods, as well as information in the research literature. It is hoped that this information will be of use to the SEMATECH members and suppliers.
Facility Fluids Metrics and Test Methods

APPENDIX A

SEMATECH Test Methods for Fluids and Fluid System Components

RTI Report No. 95C-6145/01
SEMATECH Contract # 3401550
DOC ID #: 92071220B-STD
Title: SEMASPEC Guide to Provisional Test Methods for Mass Flow Controllers
Document date: Jan 29, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This guide provides supplementary information about several test procedures available for testing mass flow controllers (MFCs). It is also intended to serve as a summary of the methods. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC test methods development task force. It does not provide detailed information sufficient for conducting the procedures, but rather discusses and cites the applicable test method or standard.

DOC ID #: 92071221B-STD
Title: SEMASPEC Provisional Test Method for Determining Accuracy, Linearity, Repeatability, Short Term Reproducibility, Hysteresis, and Deadband Thermal Mass Flow Controllers
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This test method provides a procedure for characterizing mass flow controllers (MFCs) being considered for installation into high-purity gas distribution systems. This method will quantify the accuracy, linearity, repeatability, short term reproducibility, hysteresis, and deadband of a thermal MFC. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.

DOC ID #: 92071222B-STD
Title: SEMASPEC Provisional Test Method for Determining Reproducibility and Zero Drift for Thermal Mass Flow Controllers
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This test method provides a procedure for characterizing mass flow controllers (MFCs) being considered for installation into high-purity gas distribution systems. This method will quantify reproducibility and zero drift of a thermal MFC. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.

DOC ID #: 92071223B-STD
Title: SEMASPEC Provisional Test Method for Determining Warm-up Time of Mass Flow Controllers
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This test method provides a procedure for characterizing mass flow controllers (MFCs) being considered for installation into high-purity gas distribution systems. The purpose of this method is to provide a standardized method for quantifying the warm-up time of a mass flow controller (MFC). The test conditions in this method are intended to simulate benchtop warm-up, with an MFC that has been equalized to ambient conditions for 24 hours before the application of power. Due to a wide range of manufacturing variability, warm-up times vary widely for the same model of MFC. This specification addresses a method for taking a single data point repetitively from the same MFC. Resulting data will show exactly how warm-up effects change the delivered flow of that particular MFC. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.
DOC ID #: 92071224B-STD
Title: SEMASPEC Provisional Test Method for Determining Reliability of a Mass Flow Controller
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This test method provides a procedure for characterizing mass flow controllers (MFCs) being considered for installation into high-purity gas distribution systems. The document describes a method to help determine the ability of an MFC to meet the manufacturer's published specifications over its lifetime. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.

DOC ID #: 92071225B-STD
Title: SEMASPEC Provisional Test Method for Verification of Calibration Accuracy Conversion Factors for a MFC Using Surrogate Gases
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This test method provides a procedure for characterizing mass flow controllers (MFCs) being considered for installation into high-purity gas distribution systems. The method quantifies the accuracy and linearity of an MFC when mapping an MFC's calibration from one specific gas to another. It also quantifies the flow dependence of an MFC's conversion factor. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.

DOC ID #: 92071226B-STD
Title: SEMASPEC Provisional Test Method for Determining Particle Contribution by Mass Flow Controllers
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This test method provides a procedure for characterizing mass flow controllers (MFCs) being considered for installation into high-purity gas distribution systems. The method is used to measure particle contribution by MFCs to high-purity gas systems by yielding statistically significant comparisons of particle contribution among MFCs under test conditions. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.

DOC ID #: 92071227B-STD
Title: SEMASPEC Provisional Test Method for Determining Moisture, Oxygen, and Total Hydrocarbon Contribution/Retention by Mass Flow Controllers
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This test method provides a procedure for characterizing mass flow controllers (MFCs) being considered for installation into high-purity gas distribution systems. The document describes a test method that yields statistically significant comparisons of moisture, oxygen, and hydrocarbon contamination for MFCs under test conditions. The document is designed such that tests for moisture, oxygen, and hydrocarbons may or may not be performed individually. This revision of the document incorporates changes made as a result of industry review and from corrections.
made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.

DOC ID #: 92071228B-STD
Title: SEMASPEC Provisional Test Method for Determining Mass Flow Controller Performance Characteristics from Ambient and Gas Temperature Effects
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: The purpose of this document is to define a method for testing mass flow controllers (MFCs) being considered for installation into a high-purity gas distribution system and to quantify ambient and gas temperature effects on the MFCs indicated and actual flow. The method applies to metal and polymer-sealed MFCs with flow rates up to 30 slpm. The tests include: Ambient Temperature Effects (Steady State and Transient) and Gas Temperature Effects (Steady State and Transient). This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.

DOC ID #: 92071229B-STD
Title: SEMASPEC Provisional Test Method for Determining Pressure Effects on Indicated and Actual Flow for Mass Flow Controllers
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This test method provides a procedure for characterizing mass flow controllers (MFCs) being considered for installation into high-purity gas distribution systems. This test method measures the upstream (inlet) and downstream (outlet) transient pressure influences on indicated and actual flow. The method yields the results of actual output flow versus MFC set-point and indicated flow as influenced by steady state inlet pressure. The method applies to MFCs with maximum flow ranges of up to 30 slpm. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.

DOC ID #: 92071230B-STD
Title: SEMASPEC Provisional Test Method for Determining Steady-State Supply Voltage Effects for Mass Flow Controllers
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This document defines a test method for characterizing mass flow controllers (MFCs) being considered for installation into a high-purity gas distribution system. The procedure applies to thermal MFCs. It is intended to measure the delivered mass flow rate variation as a function of deviation from the reference steady-state supply voltage. The test method is designed for DC powered MFCs. The supply voltage effects include voltage depression and over-voltage variations in the DC supply. This test method is not designed for AC-powered MFCs. This test method addresses steady-state effects and does not address any effects caused by transient power supply behavior. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.
DOC ID #: 92071231B-STD
Title: SEMASPEC Provisional Test Method for Evaluating the Electromagnetic Susceptibility of Thermal Mass Flow Controllers
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This document presents a test method that may be applied to evaluate the susceptibility of the mass flow controller electronics to electromagnetic interference (EMI). The test method covers both the radiated susceptibility and the conducted susceptibility of the controller where exposed to EMI. The electromagnetic susceptibility requirements are extracted from MIL-STD-461C and SAMA PMC-33.1, and the test method is a composite of the RS03, CS01, CS02, and CS06 test methods defined in MIL-STD 462. The test method is not designed for AC-powered MFCs. It addresses electromagnetic susceptibility of MFCs through DC power leads and control signals. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.

DOC ID #: 92071232B-STD
Title: SEMASPEC Provisional Test Method for Determining Attitude Sensitivity of Mass Flow Controllers (Mounting Position)
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This test method describes an attitude sensitivity test for mass flow controllers (MFCs). The MFC is operated during the test, and changes in calibration between the vertical and horizontal orientations of the device are monitored. This characteristic is measured at both low and high input pressure. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.

DOC ID #: 92071233B-STD
Title: SEMASPEC Provisional Test Method for Determining the Corrosion Resistance of Mass Flow Controllers
Document date: Feb 05, 1993
Descriptor(s): testing; mass flow controllers; gas distribution systems
Abstract: This test method is intended to help differentiate between mass flow controller (MFC) designs on the basis of relative resistance to corrosion-induced failure. The method describes a corrosive gas exposure test for MFCs. The test is intentionally aggravated to accelerate the test while simulating conditions that may be found within process equipment gas systems in the semiconductor industry. The MFC is operated during the test. As the relationship between corrosion and performance may differ with MFC design, corrosion is not measured directly. The effects of corrosion are detected by observing changes in MFC calibration and other operating parameters. This test is intended only for MFCs manufactured for use in HCl or a similar corrosive gaseous environment. This revision of the document incorporates changes made as a result of industry review and from corrections made during working session four of the MFC Test Methods Development Task Force. This test method is provisional until it has been validated.

DOC ID #: 92010933B-STD
Title: SEMATECH Guide to Test Methods for UPW Distribution System Components
Document date: Jun 19, 1992
Abstract: Because of the great variety of components used in ultrapure water (UPW) distribution systems and their different designs, a variety of procedures is necessary. This guide provides a brief overview of the applicable procedures for evaluating the performance characteristics of UPW...
distribution system components. The purpose for using these test methods is to enable improvements in manufacturing of UPW components, and to empower the user or purchaser of components to make better decisions regarding cost versus quality. Four categories of test method matrices are given (one for particles, two for chemical, and two for surface tests). The type of components covered by the test procedures described include: tubing and piping; fittings, regulators; valves; filter housing and cartridges; O-rings, gaskets; and ion exchange resins. The matrices show the identification number of the test method suitable for use with a given component. Each applicable test method is then briefly summarized, describing the method's intended use.

DOC ID #: 92010934B-STD
Title: SEMATECH Provisional Test Method for Sample Preparation for Chemical Testing of UPW Distribution System Components
Document date: Jun 19, 1992
Abstract: This document defines the sample pretreatment and preparation procedures for chemical testing of UPW distribution components. It includes sample preparation techniques that can be applied to the prepared samples derived from piping, tubing, fittings, valves, regulators, filter housings, filter cartridges, O-rings, gaskets, and ion-exchange resins. This test method is provisional until it has been validated.

DOC ID #: 92010935B-STD
Title: SEMATECH Provisional Test Method for Electrical Resistivity of Ultrapure Water
Document date: Jun 19, 1992
Abstract: This test method provides a procedure for determining the electrical resistivity of water. It can be used to detect ionizing impurities dissolved in treated waters prepared for electronic manufacturing facilities. It is intended for UPW components including tubing, piping, fittings, valves, regulators, filter housings and vessels, and filter cartridges, O-rings, gaskets, and ion-exchange resins. This test method is provisional until it has been validated.

DOC ID #: 92010936B-STD
Title: SEMATECH Provisional Test method for Determining Leachable Trace Inorganics in Ultrapure Water Distribution System Components
Document date: Jun 19, 1992
Abstract: This test method provides a procedure for the determination of trace inorganic impurities that leach from a water distribution system component into ultrapure water. Samples are analyzed for trace metals by inductively coupled plasma-mass spectrometry (ICP-MS) and graphite furnace atomic absorption spectroscopy (GFAAS); for cations and anions by ion chromatography (IC); and for silica by colorimetry. It is intended for UPW components including tubing, piping, fittings, valves, regulators, filter housings, filter cartridges, O-rings, gaskets, and ion-exchange resins. This test method is provisional until it has been validated.

DOC ID #: 92010937B-STD
Title: SEMATECH Provisional Test Method for the Evaluation of Bulk Polymer Samples of Ultrapure Water Distribution Systems Components
Document date: Jun 19, 1992
Abstract: This test method uses Fourier transform infrared spectroscopy to examine bulk polymer samples of UPW distribution system components. It is intended to qualitatively verify the chemical structure of a polymer resin by comparing the sample's mid-infrared spectrum with that of a reference standard. This test method is provisional until it has been validated.
DOC ID #: 92010938B-STD
Title: SEMATECH Provisional Test Method for Determining Bulk Trace Metals in Polymer Materials of Ultrapure Water Distribution System Components
Document date: Jun 19, 1992
Abstract: This test method provides a procedure for determining the nonvolatile trace inorganic impurities in bulk polymeric material used for components of UPW distribution system. It can be used with inductively coupled plasma-mass spectrometry (recommended), graphite furnace atomic absorption spectroscopy, or inductively coupled plasma atomic emission spectroscopy. It is intended for UPW components including tubing, piping, fittings, valves, regulators, filter housings and cartridges, o-rings, gaskets, and ion-exchange resins. This test method is provisional until it has been validated.

DOC ID #: 92010939B-STD
Title: SEMATECH Provisional Test Method for Evaluating Bulk Polymer Samples of Ultrapure Water Distribution System Components (DSC and TGA Methods)
Document date: Jun 19, 1992
Abstract: This test method uses thermal analysis techniques to examine polymeric samples used in ultrapure water distribution system components. Comparison of the sample to a reference standard provides indications of performance characteristics of the resin (chemical inertness, thermal stability) and physical properties of the polymer resin (crystallinity, molecular weight, morphology). The procedure uses differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA).

DOC ID #: 92010940B-STD
Title: SEMATECH Provisional Test Method for Determining the Water Retention Capacity of Ion-Exchange Resins Used in UPW Distribution Systems
Document date: Jun 19, 1992
Abstract: This test method determines the amount of water retained by hydrated ion-exchange resins. Procedures are provided for cation, anion, and mixed-bed resins. The method is applicable to both new and used resins. This test method is provisional until it has been validated.

DOC ID #: 92010941B-STD
Title: SEMATECH Provisional Test Method for Pressure Cycle Testing Filter Cartridges Used in UPW Distribution Systems
Document date: Jun 19, 1992
Abstract: This test method provides a standardized method for determining the pressure cycle fatigue characteristics of cartridge filters. It can be used to verify mechanical pressure resistance of filter cartridges, to confirm that a filter cartridge can withstand exceptional conditions of use, and to comparatively quantify the loss of mechanical strength due to exposure to exceptional conditions.

DOC ID #: 92010942B-STD
Title: SEMATECH Provisional Test Method for Determining the Flow Coefficient of Filter Cartridges Used in UPW Distribution Systems
Document date: Jun 19, 1992
Abstract: This test method can be used to verify that a filter cartridge will produce the rated forward flow at the specified applied pressure or the rated pressure loss at a specified flow rate. It applies to filter cartridges used in UPW distribution systems. This test method is provisional until it has been validated.
DOC ID #: 92010943B-STD
Title: SEMATECH Provisional Test Method for Pressure Leak Testing Filter Cartridges Used in UPW Distribution Systems
Document date: Jun 19, 1992
Abstract: This procedure provides a uniform method for detecting leakage in a cartridge filter by gas pressurization while the test specimen is submerged in a liquid of low surface tension. It is intended for verifying the assembly integrity of filter cartridges for ultrapure water distribution systems.

DOC ID #: 92010944B-STD
Title: SEMATECH Provisional Test Method for Pressure Proof Testing Filter Cartridges Used in UPW Distribution Systems
Document date: Jun 19, 1992
Abstract: This test method is used to determine mechanical pressure resistance of filter cartridges for UPW distribution systems. It is used to verify that filter cartridges will withstand their rated pressurization without integrity failure or permanent mechanical deformation. This test method is provisional until it has been validated.

DOC ID #: 92010945B-STD
Title: SEMATECH Provisional Test Method for Determining the Operational Pressure of Plastic Valves Used in Ultrapure Water Distribution Systems
Document date: Jun 19, 1992
Abstract: This test method provides a uniform method for verifying the pressure and temperature ratings of plastic valves used in ultrapure water (UPW) distribution systems by operating the valves while they are exposed to fluid media. It is intended for valves in which all parts in contact with the fluid media are plastic. It is applicable in a temperature range between 15 degrees C and 205 degrees C. This test method is provisional until it has been validated.

DOC ID #: 92010946B-STD
Title: SEMATECH Provisional Test Method for Determining the Seat Leakage of Control Valves Used in UPW Distribution Systems (Bubble Leak Detection) Detection
Document date: Jun 19, 1992
Abstract: The purpose of this test method is to provide a uniform procedure for determining the sealing characteristics across the internal sealing area (seat) of plastic valve products used in ultrapure water (UPW) distribution systems. This test method is provisional until it has been validated.

DOC ID #: 92010947B-STD
Title: SEMATECH Provisional Test Method for Leak Testing Tube Fitting Connections Used in Ultrapure Water Distribution Systems
Document date: Jun 19, 1992
Abstract: This test method provides an immersion leak test method applicable to tube fitting connections used in UPW distribution systems. It is intended for testing the integrity of the fitting connection with no externally applied load. It is applicable to tube fitting connections in which all parts in contact with the media are constructed of plastic materials. This test method is provisional until it has been validated.
DOC ID #: 92010948B-STD  
Title: SEMATECH Provisional Test Method for Determining the Hydraulic Burst Pressure of Ultrapure Water Distribution System Components  
Document date: Jun 19, 1992  
Abstract: This test method provides a procedure for determining the burst pressure of plastic components used in UPW distribution systems. It is applicable to products in which all parts that are in contact with the fluid media are plastic. This test method is provisional until it has been validated.

DOC ID #: 92010949B-STD  
Title: SEMATECH Provisional Test Method for Determining Particle Contribution and Retention by Ultrapure Water Distribution System Components  
Document date: Jun 19, 1992  
Abstract: This test method provides a procedure for determining statistically significant particle retention and shedding comparisons between ultrapure water distribution system components that are tested under representative conditions. The document contains three annexes that describe methods for characterizing and qualifying particles. The method is intended for piping and fittings, valves, pressure regulators, filters and filter housings. This test method is provisional until it has been validated.

DOC ID #: 92010950B-STD  
Title: SEMATECH Provisional Test Method for Visual Characterization of Surface Roughness for Plastic Surfaces of UPW Distribution System Components Component  
Document date: Jun 19, 1992  
Abstract: This document describes a technique for preparing test specimens of plastic ultrapure water distribution system components and defines a method for visually characterizing the prepared specimens. The document includes an appendix that contains information on other analytical techniques that may provide greater resolution of surface defects and topography (roughness). The method applies to all surfaces of plastic components used in ultrapure water distribution systems. This test method is provisional until it has been validated.

DOC ID #: 92010951B-STD  
Title: SEMATECH Provisional Test Method for Optical Analysis of Plastic Surface Condition of Ultrapure Water Distribution System Components  
Document date: Jun 19, 1992  
Abstract: This test method provides a means of testing functional surfaces of plastic components for gross surface morphology (imperfections or defects). It applies to pipes, fittings, and valves being considered for installation into ultrapure water distribution systems. This test method is provisional until it has been validated.

DOC ID #: 92010952B-STD  
Title: SEMATECH Provisional Test Method for Determining the Surface Roughness of Ultrapure Water Distribution System Components (AFM Method)  
Document date: Jun 19, 1992  
Abstract: This test method uses atomic force microscopy (AFM) to obtain three-dimensional topographic maps of surfaces of plastic components that are being considered for use in ultrapure water distribution systems. It applies to the characterization of plastic surfaces with a $Z_{max}$ (maximum height difference over entire surface) of 5 microns or less per scan. This test method is provisional until it has been validated.
DOC ID #: 92010953B-STD
Title: SEMATECH Provisional Test Method for Determining the Surface Roughness of Ultrapure Water Distribution System Components (STM Method)
Document date: Jun 19, 1992
Abstract: This test method uses scanning tunneling microscopy (STM) to obtain three-dimensional topographical maps of the surface of plastic parts being considered for use in ultrapure water distribution systems. It applies to the characterization of plastic surfaces within a Zmax (maximum height difference over entire surface) of 5 microns or less per scan. This test method is provisional until it has been validated.

DOC ID #: 92010954B-STD
Title: SEMATECH Provisional Test Method for Determining Surface Roughness of UPW Distribution System Components (Noncontact Optical Profiling)
Document date: Jun 19, 1992
Abstract: This test method provides noncontact optical techniques for measuring surface roughness of plastic surfaces with an average roughness between 0.01 micron and 1 micron. It applies to plastic components being considered for installation into ultrapure water distribution systems. This test method is provisional until it has been validated.

DOC ID #: 92010955B-STD
Title: SEMATECH Provisional Test Method for Analyzing the Plastic Surface Condition of Ultrapure Water Distribution System Components (SEM Method)
Document date: Jun 19, 1992
Abstract: This test method uses a scanning electron microscope (SEM) to test the functional surfaces of plastic components for surface morphology and for identification of inclusions on surfaces. It may be used to evaluate imperfections or defects of 0.1 micron or larger. The method applies to components such as pipes, tubing, fittings, and valves being considered for use in ultrapure water distribution systems. This test method is provisional until it has been validated.

DOC ID #: 92010956B-STD
Title: SEMATECH Provisional Test Method for Analyzing the Plastic Surface Composition and Chemical Binding of Components of UPW Distribution Systems (ESCA) Method
Document date: Jun 19, 1992
Abstract: This test method uses electron spectroscopy for chemical analysis (ESCA) to test plastic parts for surface composition and chemical bonding. The objective is to define a general set of instrument parameters and conditions that will achieve accurate and reproducible measurements of surface composition and chemical species. The test method applies to all plastic components of ultrapure water distribution systems. This test method is provisional until it has been validated.

DOC ID #: 92010957B-STD
Title: SEMATECH Provisional Test Method for Optical Analysis of Ion-Exchange Resin Beads Used in Ultrapure Water Distribution Systems
Document date: Jun 19, 1992
Abstract: This test method defines the procedure for optical testing of the physical integrity of ion-exchange resin materials (beads) used in the water purification process. The procedure uses an optical microscope at 20X magnification to determine the physical integrity of the beads. This test method is provisional until it has been validated.
DOC ID #: 92010958B-STD
Title: SEMATECH Provisional Test Method for Determining the Surface Associated Biofilms of Ultrapure Water Distribution Systems
Document date: Jun 19, 1992
Abstract: This test method is used to determine the presence of biofilms on all functional surfaces of plastic components used in ultrapure water distribution systems. It can be used either before the components are installed or after they are in place. Parts that are in service, however, may require removal and disassembly, and may, therefore, be destroyed. This test method is provisional until it has been validated.

DOC ID #: 90120390B-STD
Title: SEMASPEC Test Method for Determination of Particle Contribution by Valves in Gas Distribution Systems
Document date: Feb 22, 1993
Abstract: This SEMASPEC describes the SEMATECH test methods for obtaining statistically significant comparisons of particulate generation performance of valves and other gas distribution components under aggressive testing conditions. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTM Gas Distribution Component Test methods is published.

DOC ID #: 90120391B-STD
Title: SEMASPEC Test Method for Determination of Helium Leak Rate for Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC describes the SEMATECH test methods for leak testing components being considered for installation into a high-purity gas distribution system using a helium mass spectrometer leak detector. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTM Gas Distribution System Component Test methods is published.

DOC ID #: 90120392B-STD
Title: SEMASPEC Test Method for Determination of Regulator Performance Characteristics for Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC defines a method for quantifying regulator performance characteristics for regulators being considered for installation into a gas distribution system. Application of these test methods are expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTM Gas Distribution System Component Test methods is published.
DOC ID #: 90120393B-STD
Title: SEMASPEC Test Method for Determination of Filter Flow Pressure Drop Curves for Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC establishes a test method for preparing a flow rate vs. pressure drop curve for filters being considered for installation into a high-purity gas distribution system. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTM Gas Distribution Component Test methods is published.

DOC ID #: 90120394B-STD
Title: SEMASPEC Test Method for Determination of Valve Flow Coefficient for Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC establishes a test method for determining the flow coefficient (Cv) of a valve for use in gas service. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTM Gas Distribution System Component Test methods is published.

DOC ID #: 90120395B-STD
Title: SEMASPEC Test Method for Determination of Cycle Life of Automatic Valves for Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC establishes a test method for determining the cycle life of automatic valves utilizing static, no-flow conditions. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTM Gas Distribution System Component Test methods is published.

DOC ID #: 90120396B-STD
Title: SEMASPEC Test Method for Determination of Total Hydrocarbon Contribution by Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC establishes a method for testing components for total hydrocarbons (THC) contribution to a gas distribution system at ambient temperature. This test method also allows testing at elevated ambient temperatures as high as 50 degrees C. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used.
This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTG Gas Distribution System Component Test methods is published.

DOC ID #: 90120397B-STD
Title: SEMASPEC Test Method for Determination of Moisture Contribution by Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC establishes a method for testing components for total moisture contribution to a gas distribution system at ambient temperature. This test method also allows testing at elevated ambient temperatures as high as 50 degrees C. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTG Gas Distribution System Component Test methods is published.

DOC ID #: 90120398B-STD
Title: SEMASPEC Test Method for Determination of Oxygen Contribution by Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC establishes a method for testing components for oxygen contribution to an ultra-high purity gas distribution system at ambient temperature. This test method also allows testing at elevated ambient temperatures as high as 50 degrees C. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTG Gas Distribution System Component Test methods is published.

DOC ID #: 90120399B-STD
Title: SEMASPEC Test Method for Determination of Ionic/Organic Extractables of Internal Surfaces - IC/GC/FTIR for Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC establishes a method for testing components used in ultra-high purity gas distribution systems for ionic and organic surface residues. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTG Gas Distribution System Component Test methods is published.

DOC ID #: 90120400B-STD
Title: SEMASPEC Test Method for Determination of Surface Roughness by Contact Profilometry for Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC describes the determination of numerical values for surface roughness of components used in ultra-high purity gas distribution systems. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for
installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTG Gas Distribution System Component Test methods is published.

DOC ID #: 90120401B-STD
Title: SEMASPEC Test Method for SEM Analysis of Metallic Surface Condition for Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC establishes a method of testing interior surfaces of components such as tubing, fittings, and valves for surface morphology. Its purpose is to evaluate components considered for use in ultra-high purity gas distribution systems. Application of this test method is expected to yield comparable data among components tested for the purpose of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTG Gas Distribution System Component Test methods is published.

DOC ID #: 90120402B-STD
Title: SEMASPEC Test Method for EDX Analysis of Metallic Surface Condition for Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC establishes a method for comparing the elemental composition of normal surfaces with any defects found on the surfaces of metal tubing, fittings, valves, or any metal component. Its purpose is to evaluate components considered for use in ultra-high purity gas distribution systems. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTG Gas Distribution System Component Test methods is published.

DOC ID #: 90120403B-STD
Title: SEMASPEC Test Method for XPS Analysis of Surface Composition and Chemistry of Electropolished Stainless Steel Tubing for Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC defines a method of testing the interior surface of chromium enhanced stain less steel tubing, fittings, and valves to determine the surface composition and chemistry as a measure of the effectiveness of electropolishing. Its purpose is to evaluate components considered for use in ultra-high purity gas distribution systems. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTG Gas Distribution System Component Test methods is published.
DOC ID #: 90120404B-STD
Title: SEMASPEC Test Method for Determination of Surface Roughness by Scanning Tunneling Microscopy for Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC defines a method of testing surface features in the nanometer size range. Three-dimensional data can be obtained from a surface, which can then be used to produce a model of the surface texture or to measure surface morphology. The purpose of this procedure is to evaluate components considered for use in ultra-high gas distribution systems. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTM Gas Distribution System Component Test methods is published.

DOC ID #: 91060573B-STD
Title: SEMASPEC Test Method for AES Analysis of Surface and Oxide Composition of Electropolished Stainless Steel Tubing for Gas Distribution System Component
Document date: Feb 22, 1993
Abstract: This SEMASPEC defines a method of testing the interior surface of chromium enhanced stainless steel tubing, fittings, and valves to determine the surface composition and chemistry and thereby measure the effectiveness of electropolishing. The purpose of this procedure is to evaluate components considered for use in ultra-high purity gas distribution systems. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTM Gas Distribution System Component Test methods is published.

DOC ID #: 91060574B-STD
Title: SEMASPEC Test Method for Metallurgical Analysis for Gas Distribution System Components
Document date: Feb 22, 1993
Abstract: This SEMASPEC defines a method for determining the elemental composition and metallurgical characteristics of metal used to fabricate components for high purity gas distribution systems in the semiconductor industry. The composition and metallurgy of stainless steel may be expected to affect properties of importance to this application including surface roughness, incidence of surface defects, passivation, corrosion resistance, and welding. This procedure is to evaluate components considered for use in ultra-high purity gas distribution systems. Application of this test method is expected to yield comparable data among components tested for the purposes of qualification for installation. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTM Gas Distribution System Component Test methods is published.
DOC ID #: 93021510A-STD
Title: SEMASPEC Test Method for Determination of Particle Contribution by Low Pressure Regulators in Gas Distribution Systems
Document date: Feb 26, 1993
Abstract: This test method is designed to draw statistically significant comparisons of particulate generation performance of low pressure regulators tested under aggressive conditions. The procedure uses a condensation nuclei counter (CNC) applied to in-line gas pressure regulators typically used in semiconductor applications. It applies to automatic and manual pressure regulators of various types 1/4 in. through 1/2 in. in size. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTM Gas Distribution System Component Test methods is published.

DOC ID #: 93021511A-STD
Title: SEMASPEC Test Method for Determination of Particle Contribution by Filters in Gas Distribution Systems
Document date: Feb 26, 1993
Abstract: This document describes a test method designed to draw statistically significant comparisons of particulate generation performance of filters tested under aggressive conditions. This procedure utilizes a condensation nucleus counter (CNC) applied to in-line gas filters typically used in semiconductor applications. It applies to membrane and metal filters of various types 1/4 in. through 1/2 in. in size. This test method is currently undergoing ballot for industry standardization by ASTM. The purpose of this SEMASPEC is to provide a document that member companies can use to correlate Research Triangle Institute (RTI) test data with the test method that was used. This SEMASPEC will be superseded by its equivalent ASTM standard when the complete set of ASTM Gas Distribution System Component Test methods is published.
Facility Fluids Metrics and Test Methods

APPENDIX B

SEMATECH Facility Fluids S100
Metrics and Test Methods References

RTI Report No. 95C-6145/01
SEMATECH Contract # 3401550


Optical, SEM, and LTM methods are discussed. Particles include inorganic and bacteria.


Mathematical modeling and experimental testing of moisture contamination was done on a HCl special gas cabinet. APIMS analysis was used. (Airco/BOC Electronic Gases)


(Innovative Lasers Corporation)


This paper discusses gas system components designed for high performance, minimum particulate contamination, outgassing, and in-leakage. The components represent an integration of a low pressure drop, high flow valve with branching tees and elbows to provide a very low dead volume, ultra clean solution for nearly any configuration required in today's system designs. APIMS used. (SAES Pure Gas, Intel)


The refinements of particle counters have permitted particle counting down to a level of 0.3 µm. Recent problems in IC processing, however, have made it apparent that those unobserved particles in the sub-0.3 µm sizes are causing catastrophic "yield busts." This paper presents state-of-the-art techniques for evaluating <0.3 µm particles in fluids. It further gives examples of such problems caused by 0.3 µm or smaller particles and illustrates the difficulties one encounters in identifying and resolving such problems. Discusses SEM, TEM, AEM, ESCA, FTIR, Auger, X-ray. (Balazs Analytical Laboratory)


This paper discusses issues for both the user of UPW and the analyst who is trying to measure its quality.


The article discusses The BOC Group technical research programs relating purity in gas processing to ULSI device parameters. Programs included are effects of inert gas purity on the Ti silicide for IGFET metallization; effects of argon versus nitrogen used in silicon gate oxidation process steps; and use of the Atmospheric Pressure Ionization Mass Spectrometry (APIMS). (The BOC Group, Inc.)

Bhadha, P. M., and C. L. Cowan, “Purification to PPB and PPT Levels,” In Proceedings of the 40th Annual Technical Meeting of the Institute of Environmental Sciences, 1994, pp. 161-168. Purification technology provides physical and chemical reactions that address a wide range of impurities for many gases: silicon-resoror, dopant, etchant, reactant, and purge gases. The critical validation tests for any purification system are the effective capacity (total amount of impurities removed), efficiency of impurity removal (concentration of impurity in the purified gas), and byproducts. The paper discusses both commercially available on-line monitors and test methods developed in-house. Validation tests are compared with analytical testing by APIMS. Reactive gases including H₂, NH₃, SiH₄, HCl, and SF₆ are discussed. Analytical techniques, including dew point and FTIR with a long path length cell developed for corrosive gases will be described. (Hercules Inc., Matheson/Semi-Gas Systems)


An interferometer is used to measure the effect of the particle on the phase of the illuminating wavefronts. The magnitude of the phase signal is proportional to the volume of the particle. (TSI Inc.)


The residue after evaporation (RAE) analytical method involves slowly evaporating approximately one liter of ultrapure water, and then making a gravimetric determination of nonvolatile residue. The paper discusses a continuous monitoring technique to measure the concentration, in parts per billion, of nonvolatile residue in ultrapure water. The technique is based on the principle of determining RAE of atomized droplets. Each ultrapure water droplet leaves an ultra-fine nonvolatile residue particle whose size is related to the amount of residue impurity originally present in the ultrapure water. A Condensation Particle Counter (CPC) monitors the concentration of ultra-fine, nonvolatile residue particles. The CPC output, in particles per cubic centimeter, is calibrated to give a readout, in parts per billion by weight, of nonvolatile residue. The measurement range is 1 to 500 ppb. (TSI Inc.)


The paper discusses an experimental design for studying the effects of environmental process variables, such as temperature and pressure, on gas analyzer stability. (Praxair, Inc., Union Carbide Corp.)

This paper describes the development of continuous particle control for Ultra High Purity (UHP) bulk gases and UHP gas distribution systems. Improvements in particle sampling systems, development of a methodology for continuous measurement and analysis of particle data, improvements in condensation nucleus counter technology, and an application from a leading edge facility are described. Examples are discussed of continuous particle control in UHP gases and distribution systems at the 0.01 micron level, including 0.01 micron level control without point of use filtration at the inlet to the process level. (Union Carbide (Linde))

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In manufacturing semiconductor devices with gate-oxide layers finer than 100 Å, ultrahigh-purity gas systems must maintain particle control below the 0.01 μm level. This article describes the development of continuous particle control for UHP bulk gases and their delivery systems. Improvements in particle-sampling systems, development of a methodology for continuous measurement and analysis of particle data, improvements in condensation nucleus particle counter technology, and an application from a leading-edge manufacturing facility are described. It is shown that control to the 0.01 μm level can be accomplished without point-of-use filtration at the process tool.


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(Union Carbide)

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A prototype moisture generator is discussed which demonstrated versatility in its ability to generate moisture in the low ppb range and below. The system can be used to calibrate APIMS and other moisture analyzers at low ppb levels and below. (SAES Pure Gas, Inc., SAES Getters, SPA.)


A commercial cryogenic chilled mirror moisture analyzer (the Boulder Intertec CR-1) has been developed and tested for use with semiconductor process gases. The system has continuously reading and can be used for single digit ppb moisture levels. Evaluation showed that the instrument gives accurate and stable readings which are within the requirement specifications for gases used in semiconductor processing. (Buck Research Inc.)

Statistical, graphical, and tabular methods are used to illustrate the strengths and weaknesses implicit in some definitions of LOD (limit of detection). (Air Products and Chemicals, Inc.)


(Millipore)

This paper presents a survey of methods that have been developed for the analysis of trace organic compounds (TOC) in inorganic semiconductor processing liquids. There is also a discussion of TOC levels in selected fluids, including sulfuric acid, phosphoric acid, hydrofluoric acid, ammonium hydroxide, sodium hydroxide and hydrogen peroxide. High levels of organics (TOC > 1 mg/L) are found in many of the chemicals, especially hydrogen peroxide and sulfuric acid. (Balazs Analytical Laboratory)

This paper discusses a method for evaluating the performance of electronics grade filters in actual process chemicals. "State-of-the-art" particle monitors were used to determine the instantaneous retention characteristics of the filters. (Pall Corporation)
Carmody, J. C., A. R. Lindahl, and J. E. Martyak, "Meeting the Challenges of RO/ DI System Contamination Control in the 1990's," Microcontamination 8, Feb. (1990), pp 29. Deionized (DI) water is used at IBM's East Fishkill (EF) facility in Hopewell Junction, NY in chemical processes, semiconductor device and multilayer ceramic manufacturing, steam generation for cleanroom humidity control, cleanroom sinks for washing core area floors, walls, and tools, and for process-tool cooling. To ensure that this water meets functional requirements, the company has adopted a complete program for controlling contamination. This approach focuses on system design, system ownership, and system analysis. (IBM)


Carpenter, S., K. Hall, and Ken Saul, "Playing the Numbers Game. Achieving Accurate Bacteria Counts Using Epifluorescence." Microcontamination 8, no. 1 (1990), pp 27. In the semiconductor industry, epifluorescence microscopy is used to count bacteria. Ultrapure water has very few bacteria, thus the method is more difficult than in other systems. This article describes the method used.


Chesters, S., G. Dodd, and E. Ozawa, "Component Failure and Materials Selection for ESG Gas Systems," In Proceedings of the Microcontamination 94 Conference, 1994, pp. 579-588. The effects of electronic specialty gases on components can be detrimental from both the standpoint of safety and purity. A brief survey of users in Japan found that most component failures occurred with halogenated gases, and that valves and regulators were the most often failed components. This paper looks at the issues of purity by showing tubing lifetime to particle generation onset due to HCl. Analysis of a valve failure is used illustrate design considerations when using certain halogenated gases. (Air Liquide)

Christenson, K., and J. Zahka, "Characterization of Chemical Filter Retention Performance," In Proceedings of the 39th Annual Technical Meeting of the Institute of Environmental Sciences, 1993, pp. 225-230. Factors which play roles in controlling the particle cleanliness of process chemicals are delivery system design, system component cleanliness, feed particulate level, and filter particle retention. A technique for measuring hard particle retention by filters was proposed in the SEMATECH Provisional Test Method for Determining Particle Contribution and Retention in UPW Distribution System Components. The technique involves challenging the filter with a mixture of latex beads ranging from 0.055 μm to 0.5 μm. This paper describes the test methodology, characterizes the retention performance of the PTFE filters of 0.2 μm, 0.1 μm, and 0.05 μm ratings, and recommends modifications to the proposed method to make it more suitable for chemicals. An optical particle counter is used. (FSI Int'l, Millipore)

Clarke, P. M., R. A. Hogle, and S. M. Lord, "The Effects of Corrosive Gases on Metal Surfaces," In Proceedings of the Microcontamination 93 Conference, 1993, pp. 433-442. A FTIR spectrometer was used to evaluate chemical interactions with 316L electro-polished stainless steel, mill finished nickel-200, chemically cleaned nickel-200, and mill finished Hastelloy C-22 tubing surfaces. Purge and bakeout performances of the tubing, and the resulting impurities generated when the tubing was in contact with WF₆ and HBr, were evaluated. Specific impurities extracted from the tubing were identified. (Airco, SML Associates)


Cooper, D. W., R. J. Miller, and J. J. Wu, "Measurement with Condensation Nucleus Counters and an Optical Particle Counter in a Cleanroom," In Proceedings of the Institute of Environmental Sciences, 1991, pp. 702-711. This paper discusses the comparison of two condensation nucleus counters having different flow rates, 0.05 and 0.09 cfm. Tests were carried out in a small cleanroom under normal and reduced flow cleanroom operating conditions. Measurements were also made with an optical particle counter, and a CNC using a fluorocarbon working fluid. (IBM)

Coronell, D. G., A. D. Johnson, M. S. K. Chen, S. N. Ketkar, D. A. Zatko, and J. V. Martinez de Pinillos, "An Integrated Approach to Understanding Moisture Behavior in UHP Gas Delivery Systems: Component Testing and Computer Simulations," In Proceedings of the 40th Annual Technical Meeting of the Institute of Environmental Sciences, 1994, pp. 237-245. This paper reports on a study to improve the understanding of moisture behavior in ultrahigh-purity (UHP) gas delivery systems. The experimental aspects of the study involved a systematic acquisition of drydown measurements of stainless steel tubing and gas system components at several moisture levels. The results from these experiments were used to validate a mathematical model of the moisture dry-down phenomena. (Air Products and Chemicals, Inc.)


This report summarizes the analytical monitoring and performance data collected for twenty-six high purity deionized water systems located at the IBM Fishkill Facility. The data is evaluated with respect to the degree of treatment provided and significant events during the monitoring period. Analysis included viable bacteria, particles (by SEM), and ions (using spectrophotometer, ICP-MS, anion chromatography, chelation chromatography, TOC analyzer). (Metcalf & Eddy, Inc.)


(Research Triangle Institute)

(Ashland Chemicals)


Considerable efforts are being made to reprocess DI water and chemicals and to reduce their consumption inside fabricators. This paper discusses a dry, cleaning technology, the Radiance Process, which provides a 10% reduction in capital outlays; reduces waste disposal and water and chemical consumption; and increases the profit potential of a fabricator. (Radiance Services Company)


Recent advances in ultra-clean technology for gas distribution have led to impressive improvements in point of use purity. This paper discusses contamination transport models to simulate the convection/diffusion and adsorption/desorption of contaminants in gas distribution systems. The simulation algorithms for these components may be combined to predict the contamination performance of complete gas systems. (Airco - The BOC Group)
This paper describes the equipment and methods utilized, and the results obtained, in measurements of gas purity for the commissioning and evaluation of high purity nitrogen gas plants and other gas distribution facilities located in the United States, the United Kingdom, and Japan. The analyses used atmospheric pressure ionization mass spectrometers (API MS), reduction gas analyzers (RGA), and particle analyzers. (The BOC Group)

The paper discusses an FT-IR system specifically for measurement of moisture at levels of 25 ppb or less in selected corrosive gases of interest to the semiconductor industry, namely HCl and HBr. This system utilizes a proprietary corrosion resistant low moisture background gas cell. The paper describes the equipment and methods utilized, and the results obtained, in measurements of moisture concentrations in nitrogen, HCl, and HBr. (The BOC Group)

The experimental results reported in this paper document the capabilities of a chilled mirror hygrometer, an electrolytic hygrometer, and an API MS to measure ppb and sub-ppb moisture levels in nitrogen. (The BOC Group)


Several trace elements have been determined in photoresist by graphite furnace atomic absorption and ICP-atomic emission analysis of directly diluted resists. Detection limits and recovery levels were determined. Results were compared to ICP-mass spectrometry. (OCG Microelectronic Materials)

High-purity gases are used to establish and maintain the necessary environmental conditions in almost all chip making steps. Although nitrogen, oxygen, hydrogen, and other gases are supplied by bulk delivery systems, the remainder of the gases employed in ion implantation, chemical vapor deposition, plasma etching, and physical vapor deposition - specialty or process gases - are specially purified and packaged to ensure that the increasingly stringent requirements for ultralow contamination are met. However, ensuring that the same high purity is maintained at a customer site and in the process equipment involves an added level of technical ingenuity. This paper discusses cleanroom filling, in which cylinders are filled without degrading gas quality. (Matheson Gas Products)

(Anatel Corporation)
Acceptable levels of contaminants in water are well known for each level of circuit integration. These contaminants are analyzed in the feed water and then during primary treatment, storage, polishing, distribution and at point of use (POU). Recent research has yielded a large amount of practical information on POU contamination. The paper discusses POU organic contamination as measured by total oxidizable organic carbon (TOC) and monitoring of dissolved inorganics (ions) reported as resistivity. POU monitoring in the actual rinsing situation is emphasized, since rinse water is the most critical point for pure water in semiconductor manufacturing. (Contamination Control Research)

This paper discusses the history of the International Standards Organization (ISO) development, including the documentation required of Good Manufacturing Practice (GMP) as recommended by the Pharmaceutical Manufacturing Association (PMA) committee and the ISO for Pure Water treatment systems. (Process Equipment Unlimited, Ideal Horizons, Inc.)


Sub-micron particle analysis methods for VLSI gas distribution systems include laser particle counting and SEM/microprobe evaluation of particles collected on membrane filters. In this paper, the performance and advantages of automated SEM/microprobe methods is compared to the performance of laser particle counters in counting and sizing submicron particles. This comparison of particle counting methods involves analyses of membrane filters exposed to gas streams that were simultaneously sampled by laser particle counters. Experimental results demonstrate the ability of the SEM/microprobe technique to count, size, and compositionally identify submicron particles that are typically found in high purity gas streams. (The BOC Group)

(AMD, Andersen DeBartolo Pan, Inc.)

(Praxair, Inc., SENSAR Corp.)

This paper discusses custom designed, field qualified Gas Monitoring Systems (GMS) have been developed for continuous monitoring, data display and archiving of nitrogen contaminant loads and Atmospheric Pressure Ionization Mass Spectrometer (APIMS) technology with part per trillion detection. These new techniques make possible onsite nitrogen delivery to below part per billion levels. (Union Carbide (Linde))

This article reviews ultrapure water specifications and the contaminants of greatest concern in semiconductor fabrication. It also discusses the contaminant-removing capabilities of common types of purification equipment, as well as how each affects and is affected by other parts of the system. (U.S. Filter/IWT)

Monitored for TOC, resistivity, dissolved ozone, and particles. (University of Arizona)

This paper discusses the experimental evaluation of a new liquid-borne particle counter based on interferometry and uses bright-field rather than dark-field detection. The sizing accuracy of polystyrene latex spheres by the instrument in deionized (DI) water is presented and its response to particles in semiconductor process chemicals is compared to the response of dark-field light-scattering sensors. Its ability to measure flow rate was also determined in several semiconductor process chemicals. The evaluation verifies that the interferometric counter has good sizing accuracy, rejects gas bubbles, and is apparently noise insensitive.

Particle concentrations in semiconductor process chemicals are usually measured using light scattering instruments calibrated for use in water. Because of high vapor pressures, high particle concentrations, and refractive indices, there can be errors in data interpretation. This paper describes methodology developed to reduce the magnitude of these errors. (Millipore)

A novel dual asymmetric PTFE membrane has been developed for filtration of semiconductor process chemicals. This membrane has a dual asymmetric structure with a thin small pore sized layer sandwiched between two related open layers. In this study, the ability of this filter to reduce particle concentrations in sulfuric acid was measured and its performance compared to that of a conventional 0.1 μm stretched PTFE filter. Sulfuric acid was chosen for the testing because it contains high particle concentrations and is difficult to filter. Extensive testing was performed under ideal filtration conditions which simulated filter use in semiconductor process fabs in chemical delivery systems and in conditions which simulate point-of-use filter applications. Particle concentrations downstream of both filter types were highly dependent upon the testing mode. Steady-flow conditions gave much lower particle concentrations than conditions which subjected the filter to severe hydraulic shocks, such as point-of-use applications. The asymmetric filter provided lower particle concentrations than the conventional filter in all of the tests. The largest differences were seen for 0.1 μm-0.3 μm particles. Used IMOLV 3 sensor (volumetric spectrometer), M65 in-situ monitor. (Grant Consulting, Parker Hannifin Corporation)
Bulk chemical delivery systems can be made to deliver chemicals with low particle concentrations. However, the particle levels in the chemicals delivered is highly dependent upon how the system is designed and operated. This paper discusses how system design and mode of operation can be optimized to obtain low particle levels. (FSI International, Ashland Chemical)

Grant, D. C., D. Smith, P. Palm, F. C. Wang, D. Charest, J. Campaneria, L. S. Wai, I. Lye, K. Y. Yeo, C. K. Sing, K. Y. Tey, and D. Kaur, "Design and Certification of High Purity Delivery Systems for Semiconductor Wafer Cleaning Chemicals," Journal of the IES 37, no. 6 (1994), pp 32-40. The wafer cleaning procedures used in new semiconductor manufacturing facilities require extremely high purity chemicals. Delivering chemicals of this quality requires careful management of the chemicals from their manufacturing site to the points of use within the wafer fabrication facility. Chemical management includes proper chemical production, transportation to the wafer fab and design and operation of the chemical delivery system within the fab. This paper describes the technology used to supply 15 different types of chemicals to more than 60 POUs in the TECH semiconductor wafer fab in Singapore. The certification and continuous monitoring program confirms sub-ppb chemical delivery with particle concentrations of < 3 particles/ml at ≥ 0.2 μm. Several challenges associated with the initial design and installation of the chemical delivery system and their resolution are also described. Used ICP-MS and GFAAS for metallic ion concentrations, and a particle counter with a minimum detectable particle diameter of 0.2 μm. (FSI International, Texas Instruments, TECH Semiconductor, LaPorte Chemical)

Grant, D. C., S. Van Dyke, and D. Wilkes, "Issues Involved in Qualifying Chemical Delivery Systems for Metallic Extractables," In Proceedings of the 39th Annual Technical Meeting of the Institute of Environmental Sciences, 1993, pp. 200-208. Chemical delivery systems are required to deliver high purity chemicals to points of use within semiconductor process fabs. Prior to use these systems must be qualified to ensure that they do not add metallic impurities to the chemical. There are numerous technical issues involved in obtaining accurate measurements of the extraction rate from these systems. This paper discusses these issues and how they affected the qualification of the chemical delivery system at Motorola MOS11. Use ICP-AES and GFAAS for analysis.


As semiconductor devices become smaller and more complex, contamination control becomes increasingly crucial to ensuring defect-free products. Each material used in the fabrication line must be examined for its ability to maintain required cleanliness levels. The analyses performed will be dependent on the type of material used. In one example, electropolished stainless steel was examined for surface contamination, composition and thickness of the chromium-enriched oxide layer, and surface morphology. A second example involved analyses of plastic components
for ultrapure-water distribution systems to verify polymer identity, measure organic and inorganic contamination, and detect the presence of low levels of ionic species and very thin layers of organic contaminants. (Surface Science Laboratories)

(Alphagaz, Liquid Air Corp.)

(Ultra Clean Technology, Inc., Airco/BOC Electronic Gases)

Gruver, R., R. Silverman, and J. Kehley, "Correlation of Particulates in Process Liquids and Wafer Contamination," In Proceedings of the Institute of Environmental Sciences, 1990, pp. 312-315. This report discusses the design features and operating characteristics of corrosive chemical purification systems which can supply ultrapure chemicals. Recirculation of the chemicals was required for accelerated system cleanup and minimal chemical particulate concentrations. Chemicals meet ULSI particulate requirements. Particle concentrations for the bulk delivery were 0-10 x 10^4 per liter, while the best bottle chemicals were 10^4 per liter. Particle densities on monitor wafers showed significant improvements of 0.16 particles per cm^2 for the bulk delivered versus bottle fed chemicals. The commercial particle counter had a detection limit of 0.5 μm.
(IBM)

Gupta, P., M. Van Horn, and M. Frost, "Metal Contamination of Silicon Surfaces; Correlation of Chemical Purity to Silicon Surface Contamination," In Annual Semiconductor Pure Water and Chemicals Conference Proceedings, 1992, pp. 191-198. To determine the required level of purity needed in semiconductor manufacturing, a correlation must be made between the metallic level in chemicals and extent of surface contamination that adsorbs after being processed with these chemicals. This paper describes the use of TXRF techniques to monitor the surface contamination as a function of different contaminants in various process chemicals. The effect of different chemicals and metal impurities was studied by spiking known quantities of metal impurities in the different process chemicals. These studies revealed that the level of contamination obtained on the silicon surface depended on the process chemical used. For example, spiked ammonium hydroxide solution yielded approximately an order of magnitude higher Ni, Fe, Cu surface contamination levels than identically spiked hydrochloric acid solutions. In some cases, the surface adsorption process depended on the nature of the element; Cu from hydrofluoric acid solutions adsorbed more readily than other transition elements. (Intel Corporation)

(Ashland Chemical)

---, "Examination of Polyethylene as a Packaging Material for Electronic Chemicals," In Proceedings of the Microcontamination 93 Conference, 1993, pp. 19-28. This paper describes several methods for evaluating high density polyethylene (HDPE) resins used in packaging electronic chemicals. Ashing a polyethylene resin will remove the hydrocarbon and reveals the amount of inorganic materials in the resin. This ash consists of catalytic metals in their oxide form and catalyst support material. Analysis of the ash will show
the metals that can leach into the process chemical. Organic materials in polyethylene that can leach into process chemicals can be measured by extraction techniques. The sulfuric acid color test can quickly identify resins that cause excessive organic contamination. Identifying resins that are prone to particle shedding requires filling containers with highly pure chemicals and counting particles over time. Data is presented to illustrate the polyethylene characteristics needed for packaging electronic chemicals. (Ashland Chemical, Pacific Scientific)


Holmer, A. E., M. L. Malczewski, J. Blesener, and G. Schuemann, "Design and Calibration of a Condensation Nucleus Counter Suitable for Use in Hydrogen Service," in Proceedings of the 39th Annual Technical Meeting of the Institute of Environmental Sciences, 1993, pp. 309-314. This paper discusses the used of a condensation nucleus counter (CNC) for reactive gases, including some of the differences required in both instrumentation and calibration as well as preliminary results of a CNC for use in hydrogen. A commercially available CNC was modified in four ways to achieve the desired counting results as follows: (1) Use of a perfluorinated
compound as a working fluid. (2) Adjustment of the saturator-condenser differential temperature to optimize particle detection. (3) Operation at a slightly positive pressure to avoid use of a sample pump. (4) Addition of exit flow dilution, case purge, CNC saturator/condenser purge and proper interlocks for safety. (Praxair, TSI)

Homnick, M., K. Fulford, and S. Browne, "Assessing the Design and Performance of a Central Hot Ultrapure Water System," Microcontamination 11, no. 2 (1993), pp 21. This paper describes the design and performance of a 75-gal/min electric central ultrapure water heating and distribution system recently installed at the AT&T MOS IC production facility in Orlando, FL. Generally, the system performed above design targets for temperature variation and induced no significant increases in dissolved chemical contaminants. Moderately elevated particle counts and changes in colloidal silica composition were, however, observed in the system product. (AT&T Microelectronics)


Hurd, T. Q., "Atmospheric Effects on Particle Levels in Liquid Chemicals," In Annual Semiconductor Pure Water and Chemicals Conference Proceedings, 1992, pp. 83-105. This paper discusses experiments measuring the particle contribution of an uncontrolled atmosphere to bottled chemical particle counts. There was a difference in the particulate levels of chemicals particle counted in an uncontrolled environment as opposed to those seen when the chemical was counted in a Class 10 cleanroom. Subsequent experiments executed were designed to decouple bottle handling and sampling as possible causes of the increase in particle counts. The results of all the experiments reflected an increase in contamination levels caused by both bottle transport and atmospheric addition. A method for batch sampling chemicals, utilizing a peristaltic pump, was developed and verified by: (1) injection of particle size/count standards, and (2) direct comparison of the particle counts generated by chemical samples using peristalsis and 20 psi pressure as the methods of sample transport through the sensor. (Texas Instruments)

Hurd, T. Q., H. G. Crocker, L. H. Hall, and T. Talasek, "Particle Count Verification of Liquid Optical Particle Counters Using Known Count/Size Standards," In Proceedings of the Microcontamination 92 Conference, 1992, pp. 38-49. In this study, particle standards created by JRS and Horiba were used to check the particle counting and sizing efficiencies of six Particle Measuring Systems (PMS) IMOV 0.2-HF (0.2 micron spectrometers) and one PMS M65 (0.065 micron monitor). Using these standards, end-users can objectively evaluate manufacturer claims versus actual performance. (Texas Instruments)

Hurd, T. Q., D. Dickson, A. Turner-King, and L. Hall, "An Evaluation and Comparison of 0.05 Micron Chemical Filters," In Proceedings of the Microcontamination 93 Conference, 1993, pp. 71-85. This paper describes controlled laboratory testing to determine both the filtration efficiency of 0.05 micron filters and the amount of metals that could be extracted over time. The filtration efficiency was determined using monodispersed suspensions of 0.074 and 0.087 micron.
polystyrene latex (PSL) particles. Using monodispersed challenges has been demonstrated to be a more severe test of filter efficiency. Detection of the particles was handled with Particle Measuring Systems M65 particle counters with a lower detection limit of 0.065 microns. Correlation testing was performed with the M65's prior to injecting the challenge particles to insure accurate comparison of upstream and downstream particle levels. Analysis was provided by ICP-MS and GFAA. Nine different filters from four different manufacturers were tested for extractables and ten different filters from five manufacturers were tested for particle retention. (Texas Instruments)


Instrumental methods for quantitation of elemental and ionic contaminants in ultrapure water include inductively coupled plasma/mass spectroscopy (ICP/MS), capillary electrophoresis, and trace ion chromatography. Routine detection of single viable bacterial cells, per liter of fluid volume, represents at least 5 orders of magnitude greater sensitivity than routinely used chemical analyses at the parts per trillion level. Measurement of bacterial endotoxins, nucleic acids, and other constitutive macromolecules are impressive in terms of their sensitivity. This paper presents a review of contemporary particle and bacterial measurement methods, in use in the high purity fluids area. (Micro Assays of Vermont, IBM Corp.)


The Model 800 TOC analyzer uses membrane-based CO2 sensors and on-board storage of chemical reagents. The membrane-based CO2 sensors permits accurate TOC measurement when halogenated organic compounds or compounds containing nitrogen, sulfur, phosphorus or metal ions are present in the water sample. The sensor also has excellent calibration stability. A combination of UV only and UV/peroxydisulfate oxidation is used. Accurate TOC measurements can be made in samples ranging from ultra-pure water to water samples containing up to 50 ppm TOC. (Sievers Instruments, Inc.)


This paper discusses the advantages of high resolution ICP-MS for the analysis of HCl, HNO3, and H2SO4 acids to low ppt levels, and some preliminary results on the direct analysis of photoresists will also be presented. (F.I. Elemental)


This paper discussed the elimination of water and SiH4 and the reduction to less than the detection limits (10 ppb for water by trace moisture analyzer and 0.1 ppb for SiH4 by low-temperature photoluminescence in combination with Hall measurement by MOVPE). (NIPPON SANSO Corp.)


Ishihara, Y., T. Ikeda, T. Takasaki, H. Hasegawa, R. Fukushima, N. Miki, and T. Ohmi, In Proceedings of the Microcontamination 92 Conference, 1992, pp. 75-85. This paper discusses a conductivity measurement system with a high-pressure type in-line conductivity cell. The detection limit of this measurement systems is 1.0x10^-11 S/cm and the external leakage of in-line conductivity cell is below 1.5x10^-11 Torr l/s. The electric conductivity of liquefied hydrogen chloride (HCl) was studied and it was observed that there was a definite difference in the lapse time after HCl charge. The temperature coefficient was negative in the range from -12°C to 20°C. The electric conductivity had attained 5.5x10^-10 S/cm after repeating liquefaction. (Nippon Sanso Corp., Hashimoto Chemical Corp., Tohoku University)

Ishikawa, K., H. Mihira, and T. Ohmi, In Proceedings of the Microcontamination 92 Conference, 1992, pp. 98-106. Bulk gas purity has recently achieved ppt level at the point-of-use by implementing Ultra-Clean technologies. Using the same technologies, it is possible to study the physics and chemistry of specialty gases isolated from reactions with residual impurities. The paper discusses a Closed GC-TCD. (Tohoku University)


Kanno, Y., and T. Ohmi, "Ultraclean Gas Delivery Systems Part II: Components Key to Developing Contamination-Free Gas Supply," Microcontamination 6, no. Dec (1988), pp 23. Tohoku University has developed an ultraclean gas supply system capable of delivering high-purity gases in the sub-ppb range, i.e., no particles at the ppt level. The system is devoid of dead zone and outgassing problems, particle-free at a level of 0.1 μm diameter, and free of external leaks. (Tohoku University)


Residual silica in ultrapure water sometimes deteriorates device performance when it remains on the Si wafer surface after the drying step following the ultrapure rinsing step in the water cleaning process. Better analytical methods need to be developed for determining the concentration of both ionic and non-ionic silica in ultrapure water. Non-ionic silica concentration can be determined by deducting the ionic silica concentration from the total silica concentration. Three analytical methods were compared in terms of detection efficiency of total silica: ICP-MS, Flameless Atomic Absorption, and Ion Chromatography. There is also a discussion of the effect of ozone concentration and UV irradiation level on the ionization of non-ionic silica. (Organic Corp.)


The decrease in semiconductor line-width has necessitated the detection of smaller and smaller particle contamination in order to avoid defects. As the allowable size and total amount of particles in ultrapure water drastically reduces, however, it has become increasingly difficult to count the number of particles and to evaluate the quality of ultrapure water. This paper discusses the origin of particles: monitoring ultrapure water; particle measurement; particle counters; and semiconductor manufacturing yield and reliability.


Using carefully designed experiments, the application of APIMS can be extended to concentrations as high as 100 ppb. (Air Products and Chemicals, Inc.)


A gas chromatograph was interfaced to an APIMS. Methane at a level of 4 ppb was detected in nitrogen. This leads to the possibility of using the system for oxygen and other specialty gases. If a time of flight mass spectrometer APIMS is used, detection limits can be in the ppt region. (Air Products and Chemicals, Inc.)


This paper presents data to show the analytical capability of APIMS to detect certain impurities in gases as well as present data which will clearly show the limitations of this technique. (Air Products and Chemicals, Inc.)

Ketkar, S., A. Scott, D. Avin, R. Boyer, G. Forrest, D. Christman, M. Sullivan, J. Hughes, B. Bradshaw, and D. Gross, "Evaluation of High Purity Gas Cabinets - A Study of Moisture Dry Down and
As part of the equipment selection process for the new AMD fabrication facility being built in Austin, TX, an Automatic Gas Cabinet requirement was developed, including a list of desired components and materials of construction. Four companies submitted cabinets based on the requirements. The cabinets were then evaluated with an emphasis on purity, reliability, and safety. The tests were designed primarily to determine the dry down performance of the gas cabinets using an APIMS. The testing also included characterizing the particle emission from individual components as well as the entire process panel during various simulations of actual process conditions. (Air Products and Chemicals, Inc., Advanced Micro Devices)

As purity requirements for the process gases and chemicals used in semiconductor manufacturing continue to increase, suppliers are also challenged to improve the analytical capabilities needed to insure that products are within specifications at the point of use. The APIMS has become essential in analyzing high purity gases delivered via bulk gas distribution systems. This article reports on the use of APIMS to certify three gas systems at an operational fab. The data presented demonstrate that, although diurnal fluctuations did occur for two of the gases, all three met purity requirements as delivered to the process tools. (Air Products and Chemicals, Inc., Advanced Micro Devices)

A high pressure particle analyzer is used for the analysis of high purity silane. This paper discusses the qualification of different grades and cylinder/valve packages presently in silane service. Actual particle concentrations in prepared cylinders of silane are reported and evaluated. (Airco Electronic Gases)

To meet the demands of new ultra-clean DI water systems, a new sensor was developed using high density CCD array technology and a laser diode source of 30 mw. (Particle Measuring Systems, Inc.)

The sensitivity, resolution and sample rate of optical particle monitors, counters, and spectrometers are described and compared. Optical particle counters and spectrometers require uniform sample volume illumination. Spectrometers have the highest resolution and the greatest number of size channels. Counters may have high intrinsic resolution but have fewer number of size channels provided. Monitors are a relatively new class of instruments which do not provide uniform sample volume illumination. They are becoming widely used in liquid monitoring. Monitors are simpler, less expensive devices and are characterized as having poor resolution but providing the highest sample flow rates and delivering the largest data base. When used on fluids with normal populations having an exponential size distribution, monitors show little size distribution distortion. When modal populations or deviations from exponential size distributions
are encountered, counters or spectrometers are required. This paper discusses the use of monitors and spectrometers to characterize water quality in operating fabs. Data from seventeen fabs provide comparison within the industry as well as opportunity to compare the low resolution monitors with high resolution spectrometers under field conditions. (Particle Measuring Systems, Inc.)

Gases are used in virtually all steps of semiconductor fabrication. Gases contact the wafer directly during the various process steps and must remain pure throughout the delivery process. Gas delivery systems must meet the challenge of transporting ultrahigh purity gases through miles of tubing, thousands of components, and hundreds of gas panels without contributing contamination at the point of use. (Ultra Clean Technology)


Predictions are compared with experimental results using an APIMS. (Praxair, Inc.)

Perfluoroelastomers (e.g., Kalrez, etc.) and Fluorelastomers (e.g., Viton, etc.) are widely used as seals in semiconductor processing equipment, especially as o-ring seals in 'wet' chemical process equipment, directly contacting aggressive chemicals. Ingredients/impurities contained in the o-rings and contaminants resulting from seal degradation can leach out, contaminating the chemical process stream and adversely affecting wafer production part yields. A study was conducted to characterize and quantify the elemental, anionic, and total organic carbon (TOC) extractables from a number of different sealing materials in a simulated environment. Extractables test data on a new type of high purity sealing material is also presented. Analysis included SEM, ICP-MS, GFAA (Graphite Furnace Atomic Absorption), Ion Chromatography, and TOC analyzer. (E.I. DuPont de Nemours & Company, AT&T Analytical Services)

This paper discusses recent advancements in APIMS analysis of UHP hydrogen. Special consideration is given to the unique issues related to hydrogen analysis, namely, safety and analyzer calibration. An APIMS investigation of bulk hydrogen purification equipment and distribution systems is also discussed. (Praxair, Inc.)

This paper presents a synopsis of the state of the art for current instrumentation and a discussion of currently evolving technologies resulting from the application of APIMS. (Praxair, Inc.)
This paper discusses analysis using optical particle counters, including the particle properties observed by the device, its particle sizing resolution, counting accuracy at the point of maximum sensitivity, and its sizing accuracy. The effects of changes in these parameters on reported particle size and concentration data are discussed, along with their effects on inter- and intra-device correlations.


This paper discusses the development of a system that produces high purity gases with fewer than 0.001 particles per cubic foot, or less than one particle per thousand cubic feet of gas. This level of gas purity requires continuous counting with a CNC of 0.05 cfm for fourteen days to produce one single count. The approach to the design of the system, the methods used to evaluate its performance, and the application of the system to low level background noise count studies of LPC and CNC are described. (University of Minnesota)

Methods to measure UPW at point of use include microscopic inspection of filtered samples, bacteria counts, measurement of residue on wafers, and periodic sampling with a particle counter of the liquid baths. Instrumentation is available to measure particles of certain size ranges on a continuous basis. This technique can readily provide information on the transient behavior of particle populations at selected locations in the water supply. Within the limits of detectability of the counter, a measure of the transient particle release due to pressure fluctuations can be made. With an appropriately selected time interval, a useful determination of the "clean up time" of a new filter element installation can be determined, providing a basis for specifying certain filter changing procedures. It has been found that the simultaneous use of two liquid particle counters can readily provide a measure of the effectiveness of specific final filter elements supplying a wet hood or rinser dryer. (Motorola, Inc.)

This paper examines the use of carbon adsorption technology for the recovery of 1,1-dichloro-1-fluoroethane (HCFC-141B). A gas chromatograph was used to monitor the outlet stream of the carbon bed for breakthrough of the solvent. Gas samples were collected during the desorption period to monitor the formation of breakdown products that may form when steam comes into contact with HCFC-141b. The samples were analyzed by GC/MS. (Allied-Signal, Inc.)

(NOW Technologies)

Lynn, S. Y., B. Huling, and M. Su, "Characterization of Gaseous Contamination in a Multi-Chamber Etch Tool," In Proceedings of the Microcontamination 93 Conference, 1993, pp. 113-117. Equipment design and manufacturing processes can play major roles in controlling wafer contamination. The design of multi-chamber process tools and the accompanying process is especially critical, as the wafer is typically sequenced through several different environments while undergoing sequential process steps. This paper characterizes some of the gaseous contamination found within a multi-chamber tool, it is demonstrated that the contamination can be minimized by the addition of post evacuation cycles, balanced transfer pressures between pumped modules, and carefully selected purge rates which effectively exclude contamination from entering the processing module. (Air Products and Chemicals, Inc., Lam Research Corp.)

Ma, C., A. M. Haider, and F. Shadman, "An APIMS Study on the Baking and Purging Schedule of Metallic and Ceramic Filters," In Proceedings of the Microcontamination 93 Conference, 1993, pp. 571-579. An APIMS was used to analyze moisture and oxygen impurity outgassing of metallic and ceramic particle filters. The baking/operating temperature of the filters was 200 degrees C. A model for filter analysis was applied to the experimental data to extract more fundamental information about impurity desorption from filters. Purge schedule for the particle filters at different temperatures was also obtained. (University of Arizona)

--, "Use of APIMS to Characterize Trace Impurity Distribution in Gas Delivery Systems," In Proceedings of the Microcontamination 92 Conference, 1992, pp. 174-180. Recent research on micro-contamination control has shown that Atmospheric Pressure Ionization Mass Spectrometry (APIMS) is the most sensitive and powerful analytical tool for the trace impurity detection in ultra-pure gas delivery systems. This study focuses on using APIMS to characterize trace impurity in gas delivery systems. Contamination reduction by using of Reactive Filter Purifier (RFP) and various contamination sources in gas delivery system are also discussed. (University of Arizona)

Ma, C., N. Verma, E. Shero, and F. Shadman, "An APIMS Study of Back Diffusion of Moisture and Oxygen," In Proceedings of the Microcontamination 94 Conference, 1994, pp. 406-412. Moisture and oxygen back diffusions were measured using an atmospheric pressure ionization mass spectrometer (APIMS) in ppb and sub ppb levels. Back diffusion refers to diffusion of impurity from the high concentration to low concentration against a purge flow. Samples under the study were 316L stainless steel tubes and particle filters. A theoretical model was fitted to the experimental data to generate fundamental parameters such as surface diffusion coefficient for the species. Predicted moisture back diffusions for different sizes and purge flow rates are given in this paper. (University of Arizona)


(SEMATECH)


In this study an APIIMS system was used to measure the purity of nitrogen and argon bulk gas distribution systems. Measurements were made in the purifier room and in the fabrication facility. (Air Products and Chemicals, Inc., Motorola, Inc.)

(Motorola)


This paper presents the details of several studies on biofilm growth, and evaluates the current assumption on biofilm attachment in deionized water piping systems. The studies conducted include the evaluation of laboratory methods to detect the presence of biofilms and viable bacteria. (IBM, MicroAssays of Vermont)

Biofilm, the microbiologically induced fouling of an aquatic system, occurs in deionized water systems at the interface between the liquid and the solid component wall. In the past, common industry practice has been to maintain a turbulent flow in the system to prevent such buildup. IBM conducted studies to find whether different piping materials and varying rates of flow better prevented bacterial biofilm formation. Effects of regular ozonation, point-of-use filtration, and dynamic hydraulic operations on microbiological growth were also investigated. (IBM, MicroAssays of Vermont)

Analyses include resistivity and temperature (resistivity meter and cell, temperature compensated), dissolved oxygen (oxygen meter and probe), TOC (organic monitor and probe), viable bacteria (agar growth and epifluorescence microscopy), total bacteria (SEM), pyrogens/endotoxins (LAL assay), anions (ICP/MS), cations (IC), SiO2 (ammonium molybdate method), particles (photocounting counter, laser-based counter, SEM), particle identification (energy-dispersive x-ray analysis), radiation. (IBM)

This high-level performance of the deionized water production, distribution, and polishing systems at IBM-East Fishkill requires routine and nonroutine analytical characterization. The article discusses the analytical techniques, DI water quality data, contaminant characterization, data interpretation and sampling schedules, and outside laboratory services that the IBM-EF team uses to achieve its goals. Four case studies are presented in which the various data set information and analyses have been employed to solve specific production problems. (IBM)

Particle metrology in this investigation included both standard enumeration techniques and specialized laboratory analyses. On-line particle enumeration studies utilized laser-based particle counters. The particle investigation was based on scanning electronic microscopy (SEM/EDS, 0.08 μm-5.0 μm), transmission electron microscopy (TEM/EDS, 0.01-1.0 μm), and Auger electron spectroscopy (AES, approx. 2 nm). Particle metrology in this investigation also included microorganism identification. Aerobic heterotrophic bacterial cultures were conducted using mTGE broth, R2A agar, and Pseudomonas isolation agar. Culturing was done at different media concentrations and utilized various incubation procedures. Epifluorescence microscopy and Limulus amebocyte lysate assays were performed during each sampling event. The microorganisms in the deionized water were then identified by a computerized database utilizing fatty acid profiles generated by gas chromatography. Samples were collected for heterotrophic and Pseudomonas bacterial analysis from several points within the same pipe circumference to profile the flowing deionized water distribution piping system. (IBM, Industrial and Environmental Analysts, Inc.)


Hydrofluoric acid (HF) is known for its important role in wafer manufacturing. Papers have been written which address the importance of HF chemical purity and the deposition of impurities on silicon wafers. Other studies have focused on the mechanisms by which metallic impurities in HF deposit on wafer surfaces or cause surface roughness. In 1989, Athens Corporation developed an ion exchange system to purify HF and Texas Instruments Inc. reported a 5% improvement in DRAM manufacturing yield using ion exchange purified HF. What has not been addressed is the merging of HF chemical generation technology to produce and maintain assay of very pure HF solutions, with HF ion exchange technology to prevent impurity build up in the HF solutions. This paper addresses the on-line application of the two technologies in a wet station. (Legacy Systems Inc.)

In this study ICGs (in-situ chemical generation) for SC1 and SC2 solutions are prepared by bubbling anhydrous hydrochloric acid and ammonia gas into ultrapure water. Ozone gas is used as a substitute for hydrogen peroxide. (Intel Corp.)

(Air Liquide)

Gas distribution systems for the electronics industry are required to meet ever-increasing standards of purity. This has led to considerable refinement by manufacturers of components in materials of construction, surface finish, dead volume, etc. To test the effectiveness of such refinements, it is logical to examine the response of components to impurities. In this way, we determine directly whether desirable performance is being achieved. We can also gain insight into the relationships between the nature of a component and its performance. We have developed the capability to control time dependence of impurity concentrations at low ppb levels. This has been applied to experiments designed to study the interaction of water and oxygen with
valves and mass flow controllers. The results are interpreted in terms of the properties of the components tested. Commercial oxygen and moisture analyzers are used.
(American Air Liquide, Applied Materials Inc.)

(Air Liquide)

Comparison of electrolytic hygrometer moisture analyzer and an APIMS to measure moisture using a moisture "pulse generator". (Air Liquide)

This paper discusses methods for evaluating gas panel performance with respect to moisture, particles, and removal of reactive gases. The gas panel test builds upon existing methodology established for testing individual components, and is part of a larger program to extend these component test methods to systems. The test procedure examines particle generation in various phases of operation and if these particles will be delivered to the process. It determines the moisture intrusion during cylinder changes and the factors contributing to this intrusion. It evaluates the efficacy of panel purge cycles in removing a controlled moisture input and process gases. (Air Liquide, Texas Instruments)

This paper describes a development program to ensure the highest quality of ULSI grade inert gases. Topics include selection and verification of advanced particle counters, development of accurate sampling systems, and laboratory performance testing of 10-inch filter cartridges and 1 1/2-inch valves. A unique state-of-the-art particle counter, capable of detecting particles as small as 0.003 micrometer (30 Angstroms) at a sample flow rate of 2.5 cm³/sec is briefly described.
(Air Products and Chemicals, Inc.)


To improve ultrapure water, the additional effect of HF in pure water was investigated. Both formation of native oxide on the Si wafer surface and generation of viable cells in pure water can be prevented by using pure water containing HF which was not contaminated by heavy ions and viable cells can be fed to point of use without a circular line such as a conventional pure water feed line. (Morita Chemical Industries Company Limited)

The installation of a point-of-use, in-line gas purifier does not automatically guarantee the purity of gases inside the process chamber. A commercially available resin-based purifier was characterized for three different grades of gases commonly used in semiconductor production. The results of the experiment showed that although the purifier was efficient in removing oxygen, moisture, and carbon dioxide from the three feed gases, it did not significantly reduce other impurities in the various gas samples, including carbon monoxide, methane, ethane, hydrogen, or methylene chloride. Under certain conditions, the purifier actually added small amounts of impurities, such as butane or other organics, to the gas stream. The research also pointed to the need for further studies of purifiers based on different chemistries. (Intel Corp.)

The paper discusses a new system to measure the moisture concentration in specialty gases, using an APIPS. SiH₄ and HCl gases were used for the moisture measurement experiment. (Tohoku University)

Dilute hydrofluoric acid (HF) is used to control silicon oxide thickness in controlled etch processes and to remove contaminated silicon oxides. This paper describes the design and performance of systems used to blend HF and DI water to make 0.49% and 2.5% by weight HF. The performance of two blending systems and three chemical delivery systems installed at TECH Semiconductor is described in terms of assay control and chemical purity. Several problems
associated with the initial design were encountered during system certification. Once certification was completed, the systems provided extremely pure chemical with precise concentration control. The blended chemical typically contained approximately 4 ppb total metallic ions and 0.02-0.10 particles/ml. (FSI Intl.)


Ohki, A., Y. Mizuguchi, and T. Ohmi, "Quick Inspection Technology of External Leakage for Total Gas Delivery System by APIMS," In Proceedings of the Microcontamination 94 Conference, 1994, pp. 298-307. The paper discusses a high speed inspection method developed by Tohoku University and OSK to confirm external leakage is within the value of guarantee for nonexternal gas delivery systems. The method uses an APIMS. (Tohoku University)

Ohmi, T., Y. Kasama, K. Sugiyama, Y. Mizuguchi, Y. Yagi, H. Inaba, and M. Kawakami, "Examining Performances of Ultra-High-Purity Gas, Water, and Chemical Delivery Subsystems," Microcontamination 8, no. 3 (1990), The paper discusses ultra-high-purity gas, water, and chemical delivery subsystems. The design targets for the ultra-high-purity gas line are given along with factors relating to system design and construction. Technology levels for an ultra-high-purity gas system are outlined. (Tohoku University)


Pepper, I. L., K. L. Josephson, R. L. Bailey, M. D. Burr, S. D. Pillai, D. L. Tolliver, and S. Pulido, "Measuring Bacterial Contaminants in Ultrapure Water. A Rapid Analytical Method," Microcontamination 12, no. 10 (1994), This article describes the use of a molecular biology technique known as the polymerase chain reaction (PCR) that provides rapid and sensitive detection of microbial contaminants. By employing this technique on UP-water samples collected over an 11-month period, we demonstrated that microbial contamination was episodic in nature and that 44.1% of the samples tested showed a positive indication of bacterial contamination of at least 1 CFU/L. The technique has high sensitivity, is cost-effectiveness, and has good speed of detection. (University of Arizona)

The contaminant contribution of such process-related systems as high-purity-gas distribution lines has become an increasingly important concern of the semiconductor industry since contaminant sources, including the gas itself, having been tightly controlled. This article discusses SEMATECH's development of test methods for cleanroom gas-handling systems, focusing on the test method validation studies and data generation work. (Research Triangle Institute, SEMATECH)


Standardized test methods developed by SEMATECH were used for the evaluation of the contamination performance such as particle, moisture, and total hydrocarbon emissions from ultra high purity mass flow controllers (MFCs) used in cleanroom gas delivery systems. The SEMATECH test methods were initially subjected to a validation study to correct deficiencies in the written test methods. The test methods revised after the validation study were used to generate test data that benchmarked the MFCs tested. Results from this study are presented to demonstrate the usefulness of SEMATECH's test procedures. Particle and gaseous emission data from 28 MFCs from seven suppliers are summarized. (Research Triangle Institute, SEMATECH)


This paper summarizes the development of standardized test methods by SEMATECH to verify the cleanliness, mechanical integrity, performance, and surface finish of high purity, electropolished 316L stainless steel, clean room gas handling components; and the application of these test methods to generate data on the contamination and performance of typical gas handling components. The objective was to provide data that semiconductor manufacturers can use as a guide in selecting components for their equipment and facilities. (Research Triangle Institute, SEMATECH, Pall Corporation)


This paper describes the design and fabrication of two types of solid state moisture sensors, and discuss the results of an evaluation of the sensors for the detection of trace levels of moisture in semiconductor process gases. The first sensor is based on surface acoustic wave (SAW) technology. A moisture sensitive layer is deposited onto a SAW device, and the amount of moisture adsorbed on the layer produces a proportional shift in the operating frequency of the device. Sensors based on this concept have excellent detection limits for moisture in inert gas (100 ppb) and corrosive gas (150 ppb in HCl). The second sensor is a simple capacitor structure that uses porous silicon as a moisture-sensitive dielectric material. The detection limits of these porous silicon sensors for moisture in inert gas are about 700 ppb prior to HCl exposure, and about 7 ppm following HCl exposure. (Sandia National Laboratory)


Analysis of volatiles was done using RGA and APIIMS. Particles were measured using a CNC and a LPC. (Millipore Corporation)
Plante, W., J. Jaillit, and R. Tawfik, "Using All-Stainless-Steel Filters in Hydrogen Chloride Gas Lines: An Investigation of Contamination Effects," *Microcontamination* 11, no. 5 (1993), pp 29. The paper discusses the effects of exposure to hydrogen chloride on stainless-steel filters and on all-metal tubing. Evaluation techniques were used to monitor volatile impurities in the gas stream and to assess the components' particle-shedding behavior, surface condition and chemistry. (Millipore Corporation)


---. In Proceedings of the *Microcontamination* 93 Conference, 1993, pp. 443-452. This paper discusses system purge techniques, gas filtration, and other contamination control strategies and the impact of system design and operating conditions on particle formation in silane. It also discusses the conditions under which these particles form. Analysis provided by a flow-cell type specialty gas particle monitor, and a FTIR spectrometer. (Millipore Corporation)


Rath, H. J., and R. Neunteufel, "Chemical Analysis Techniques for the Determination of Metallic Trace Impurities in Process Liquids and on Silicon Wafer Surfaces," *The Electrochemical Society Proceedings of the Satellite Symposium to ESSDERC 90 - Analytical Techniques for Semiconductor Materials and Process Characterization, Berlin Sept 1989 90*, no. 11 (1990), pp 335-352. Liquid chemicals for today's integrated circuit manufacturing are specified with regard to metal contamination in the lower ppb range. Three techniques for measuring trace metallic impurities are described, as well as a new analysis technique called vapor phase decomposition / atomic absorption spectrometry (VPD/AAS) to detect surface contamination. The sensitivity of this method offers the possibility of measuring face impurities in the range of about 10 ppm of a monolayer of silicon. Results about the deposition of several metals on silicon surfaces treated with different cleaning solutions are discussed. (Hoechst AG)

Reath, M., J. Brannen, P. Bakeman, and R. Lebel, "Use of Residual Gas Analysis in Low Pressure Semiconductor Process Reactors," In Proceedings of the Annual Meeting of the Institute of Environmental Sciences, 1993, pp. 119-123. This paper discusses the use of residual gas analysis (RGA) for troubleshooting of TEOS and tungsten chemical vapor deposition processes. In each process, RGA identified reactor impurity sources later proven to be the root cause of film defects and foreign material deposition. RGA verified the effectiveness of modified reactor hardware and operating procedures. (IBM Technology Products)

This paper discusses a test method using electron spectroscopy for chemical analysis (ECSA) to test plastic parts for surface composition and chemical bonding. The objective is to define a general set of instrument parameters and conditions that will achieve accurate and reproducible measurements of surface composition and chemical species. This test method applies to all plastic components of ultrapure water distribution systems. This test method is provisional until it has been validated. (SEMATECH)


This paper discusses the LOD (Limit of Detection) concept as it applies to the determination of ultra-trace impurities in gases. Various methods used to determine LODs will be differentiated. Factors such as linearity of response, the presence of background signals, calibration and extrapolation will be discussed using examples to show how these factors affect the values determined as LODs. Using guidelines presented in this paper, the analyst will be able to determine LODs that are realistic for the analytical method being evaluated. (Air Products and Chemicals)


The paper discusses the feasibility of using APIMS as a C1-C3 hydrocarbon monitor was investigated by Collision-Induced Dissociation (CID) of parent molecular ions in the declustering region of the APIMS source. Mass spectra were obtained for nitrogen containing low ppb levels of methane, ethane, propane, and carbon monoxide. (Air Products and Chemicals)


This paper discusses the results of an investigation involving direct analysis of tetraethoxysilane (TEOS) vapor by ICP-high resolution MS. TEOS is used as the preferred reagent for CVD deposition of SiO2. In this study, sampling by a double focusing, high resolution mass spectrometer provides separation and quantitation of elements of interest in the presence of matrix species which would obscure those elements using conventional low mass resolution methods. With the combination of resolution and accuracy in mass determination, the instrumentation has allowed, for the first time, the identification of interfering matrix species. Quantitative analysis of the volatile impurities was established and comparisons made to the nonvolatile impurities demonstrating the importance of direct analysis. (AT&T)


This paper discusses a quadrupole residual gas analyzer (RGA) package for in situ, real-time monitoring implemented on a low temperature epitaxy ultra high vacuum chemical vapor deposition reactor (LTE UHV-CVD). Various operating modes for the in-situ monitoring equipment are detailed, such as qualifying process gases and equipment, optimizing process parameters, detailing growth chemistries, real-time monitoring of out-of-spec events, and pinpointing contamination problems. (Aireo, IBM)
Rowe, R. K., B. R. Stallard, L. H. Espinosa, and T. M. Niemezyk, "FTIR Spectroscopy for the Determination of Water in Corrosive Gases," In Proceedings of the Microcontamination 94 Conference, 1994, pp. 112-120. Presently there are several technologies costing $50k or less that are capable of detecting trace water vapor as low as 50 ppb in nitrogen. However, no one type of instrument has achieved universal acceptance. In particular, all have limited compatibility with corrosive gases such as HCl and HBr. The goal of this project is to develop an in-line instrument based on infrared spectroscopy for this purpose. Earlier results showed conclusively that FTIR spectroscopy can be successfully used for trace water detection. (Rio Grande Medical Technologies, Inc., Sandia National Laboratories, University of New Mexico)

Sanborn, W. A., A. M. Brzychey, E. Flaherty, S. Muller, R. Smith, and B. Twombly, "Particle and Chemical Characterization of Clean Room Prepared and Filled Compressed Gas Cylinders," In Proceedings of the Microcontamination 93 Conference, 1993, This paper describes data obtained during the preparation, evacuation, and filling of compressed gas cylinders in a clean room environment and during the subsequent withdrawal of the gases from the cylinders. After preparation, each cylinder in this study was filled with helium. Chemical, particulate, and metals impurities were monitored for each cylinder at full pressure, half pressure, and residual cylinder pressure. Results show lower particle counts, lower metals concentration and lower moisture impurity levels for the clean room prepared and filled cylinders. (Matheson Gas Products, Inc.)

Schmidt, H. F., M. Meuris, P. W. Mertens, S. Verhaverbeke, M. M. Heyns, and K. Dillenbeck, "Evaluating the Effects of Chemical Purity within the RCA Wafer-Cleaning Process," Microcontamination 11, no. 9 (1993), pp. 27. Ultraclean processing has become a major focus in the semiconductor industry's efforts to control defect densities as device dimensions continue to shrink. Wet chemistry cleaning is a particular area of concern because of its recognized sensitivity to the quality of the chemicals employed. This article reports on a study of the correlation between the stability and performance of cleaning solutions based on hydrogen peroxide and also discusses the impact of metallic and particulate contamination and surface roughening. (IMEC, Ashland Chemicals)

Schmidt, M., "Metal Ion Control in Ultrapure Liquids: Must We Replace Stainless Steel?," Microcontamination 8, no. Dec (1990), pp. 52.

Schmit, G., Y. E. Li, J. McAndrew, and H. C. Wang, "Evaluation of Air Liquide Gas Panel for Particles, Moisture and Live Gases," In Proceedings of the Microcontamination 93 Conference, 1993, pp. 454-464. Automated cylinder gas panels are designed to deliver specialty process gases safely and to minimize contaminant intrusion during cylinder change. Air Liquide has characterized its gas panels to better understand their performance during and after the normal cylinder change purge routine. The gas panels have been characterized for moisture intrusion (during cylinder replacement), particle generation (during the purge routine) and for safe process gas removal (a live gas test). Analysis with CNC particle counter, Meeco Aquamatic+ moisture detector, and FTIR spectrometer. (Air Liquide)

This paper gives a brief survey of the effects of moisture on semiconductor processing. Examples are drawn from the open literature to highlight the following processes: physical vapor deposition (PVD), plasma etching, chemical vapor deposition (CVD), particle formation during pumpdown, and contamination in process gas tubing. An effort was made to include work which found experimental evidence of a correlation between measured moisture levels and particulate contamination or thin film properties. The paper also discusses the SEMATECH project on moisture sensors for defect reduction. The project objectives, sensor requirements, selection, and applications in plasma etch equipment are presented. Two moisture measurement techniques, Residual Gas Analysis (RGA) and capacitive hygrometry, are discussed in the context of their application as in situ moisture sensors. (SEMATECH, Intel Corp.)

The paper discusses development of a valve that can detect reverse flow rate at less than several cc per minute under less than one second response time. In the event that a 5 CCM reverse flow occurs, this check valve can shut in 0.4 seconds. The valve meets the requirements of the UltraClean gas delivery line and can be used in low flow rate CVD, etching, and ion implant processes. (STEC Inc.)

(BOC Group Electronic Gases)

This paper discusses mathematical models that simulate the transport of moisture in gas piping systems which were developed and testing using an APIMS. These models are used to predict the moisture contamination performance of various systems at the design stage. Also discussed is a technique for measuring the moisture absorption behavior of stainless-steel tubing surfaces.
(BOC Group Electronic Gases)

This paper discusses Atmospheric Pressure Ionization Mass Spectroscopy (APIMS) as a quantitative technique for analysis of gas phase impurities in ultrapure semiconductor processing gases at the parts-per-trillion (ppt) level. Improvements have been made which have lowered detection limits of APIMS to low-ppt levels and have improved the accuracy of analysis for impurities in ultra-pure nitrogen, argon, and hydrogen. The APIMS system used in these studies have been calibrated over wide ranges of impurity concentration, with data extending well into ppt ranges. The APIMS has been used for qualification and certification tests on ultra-high purity cylinder products, assemblies of gas handling components, large-scale inert gas purifiers, and state-of-the-art distribution systems. (BOC Group Electronic Gases)

This article describes the use of APIMS in the certification testing of a new distribution system, which was made of high-quality all-metal components and used state-of-the-art construction principles. The tests show consistent sub-parts-per-billion impurity levels of nitrogen delivered to the point of use. Also, a previously unreported mechanism for contamination transport in gas distribution systems was identified, which is eliminated through the dead leg free design of the ultraclean system. (BOC Group Electronic Gases)


This paper discusses the development of models which can be used to predict the contamination response of a gas piping system under varying conditions of concentration, flow rate, temperature, and pressure. The modeling package can be used as a design tool, predicting the contamination performance of a system before it is built. A system contamination transport model requires a database of component response characteristics which must be measured using test protocols specifically designed to measure the relevant parameters. Used APIMS analyzer. (Airco)


This paper discusses a project which used the APIMS and demonstrated that hydrogen with total impurity levels below 10 ppb can be delivered to the point-of-use without purifiers. (Airco, MCNC)


Liquid particle counters are now routinely used to obtain particle counts in DI water and process chemicals used in semiconductor processing. Typically, these counters run the liquid past a laser beam and sense particles by light each particle reflects or scatters. The problem is the high number of hard-to-predict variables encountered during particle counting. These variables are encountered during particle counting and make particle counts obtained with these instruments somewhat suspect. (Semiconductor Intl.)


The semiconductor industry has achieved better purity in bulk and specialty gases and in the distribution system that delivers the gases to the process tool. That trend will continue. However, there is a new focus on gas cost-of-ownership, and that raises the old question of putting very clean gases into what is basically a dirty reactor. New gas modeling programs relate gas impurity to material and component selection and defect density.


The paper describes Mattson Technology's approach to achieving high-reliability levels in semiconductor capital equipment, with MTBF's (mean time between failures) in the hundreds of hours. The program is explained, including equipment design guidelines, supplier selection criteria, life test methods, data gathering and reporting formats, corrective action, change control, equipment user training, and equipment maintenance.


This paper discusses and identifies chemicals commonly used during feedwater pretreatment; for filter and membrane cleaning, lubrication, and wetting; for regeneration of ion-exchange resins; and for deoxygenation of high-purity water. Impurities that may be added to the water from chemical residuals or generated via interactions between the various chemicals and purification system components are also described. Future trends and requirements for liquid chemical usage are discussed.

(Siltec Silicon)


This paper discusses recent experimental results on the development and evaluation of a prototype gas purifier endpoint monitor, which utilizes reactive quartz crystal microbalance technology. The technology has also been used in detecting impurity breakthrough from inert and HCl gas purifiers. (Millipore Corp., Novapure Corp.)


Aerosol contamination monitoring is a key measure to improve microprocessor performance and reduce the number of failing microcircuits due to particulate contamination. This paper addresses the most important performance parameters and specifications of optical aerosol contamination particle counters (OACPC). (Hiac/Royco)


Condensed Nucleus Counters (CNC) are used to monitor nitrogen, argon, and air in ultra-clean semiconductor manufacturing environments. With the demand for cleaner process gases, there is a need to adapt CNCs for monitoring nanometer size particles in oxygen and hydrogen. The traditional CNC working fluid, n-butyl alcohol; with MULTIFLUOR, an inert, fully fluorinated fluorocarbon has the potential to extend application of the CNC counters to reactive process gases. (Met One, Inc., Air Products and Chemicals, Inc.)


A microprocessor controlled system has been developed to generate a known level of moisture and is capable of changing both the flow rate and the temperature of the moisture source and therefore producing water vapor from 1 ppb up to 1 ppm. The system has been correlated with the response of a Fisons API 200 and that of an electrolytic cell. Data of the lifetime of an electrolytic cell with some theoretical considerations are also given. (SAES Getters S.P.A.)

Impurity analysis in the below ppb region was performed using a GFAA, an ACP-MS, and a liquid ion chromatograph. A particle counter was also used. (Santoku Chemical Inc., Tohoku University)


This paper will report empirical gas analysis and metallurgical data obtained from existing HBr systems in a production fab environment. Conclusions will be drawn which allow for the informed selection of gas purity and component specification based on cost and contamination. FTIR spectroscopy was used for gas phase analysis. Surface analytical techniques included SEM, EDS, and auger spectroscopy. (Praxair, SEMATECH, Millipore)

(Balazs Analytical Lab)


This paper discusses two new in-situ analysis techniques which are suited to accurate and responsive analysis of moisture and particles: FAST moisture analysis system and FAST particle data acquisition technique. (Aircor, BOC Group)


This paper discusses a development program to ensure the high quality of VLSI grade inert gases, including selection and verification of advanced particle counters, and development of accurate sampling systems and methods for generating pressurized test aerosols. Experimental results are presented for filter cartridges and mult cartridge filter installations obtained in a large-scale laboratory pressurized test loop and at field locations.

This paper discusses an in-line monitor for moisture based on capacitive hygrometers. The monitor has the following properties: (1) sensitivities into the single digit ppb range, (2) fast response time, (3) stability of the sensor has been increased to longer than one year, (4) retrofittable into existing process gas lines, (5) electronic functions integrate easily into most process controls. The work was carried out in nitrogen, but HCl gas will also be studied.
(Novapure Corporation)


This paper discusses the analysis of three types of valves. Particle release rates of the valves were measured using a liquid particle counter. Particle concentrations were measured initially and after 500 and 1000 cycles. Results showed that as-manufactured valves released more particles than the precleaned valves throughout the 1000 valve cycles; precleaning the valves reduced the initial particle release concentration by over 65%; and that particle release concentrations from precleaned valves were indistinguishable from the background concentration of the deionized water after 500 cycles.

Scanning electron microscopy combined with energy dispersive x-ray spectrometry is the technique of choice for particle characterization, but is slow. Automation of the system for particle detection and elemental analysis allows for detection, sizing and characterization of hundreds to thousands of particles isolated from a pure water system without human intervention after initial setup. Automated systems commonly in use provided accurate sizing and compositional information, but are limited to particles larger than approximately 0.5 μm, and rely on the operator to anticipate possible particle types (e.g., stainless steel, silica). Improvements anticipated for the near future include a smaller minimum size, ultimately approximately 0.05 μm by use of transmission electron microscopy, and a posteriori particle classification by statistical techniques. (Fluoroware, Inc.)


This paper discusses the mechanism and kinetics of back-diffusion and the dominant parameters which determine its magnitude and significance. A model was developed to simulate the process and calculate the extent of back-diffusion in a tube. Experimental and theoretical results for the
back diffusion of impurity in nitrogen carrier and the effect of impurity leakage on wafer contamination are discussed. (University of Arizona)


The paper addresses sealing technology, wet and dry processing, and supportive data that indicates how O-rings are linked to contamination, and how to control that problem. Analysis included SEM, ICP-MS. (Fluid Handling Group)


This paper describes the technology used to supply 15 different types of chemicals to more than 60 POUs in the TECH Semiconductor wafer fab in Singapore. The certification and continuous monitoring program confirms sub-ppb chemical delivery with particle concentrations of <3 particles/ml at 0.2 μm or greater. (Texas Instruments)


This article discusses the need of performance-based selection criteria and test methods that measure impurity transfer functions; the evaluation of transfer functions by several perturbation techniques, using moisture and particles as examples; and the kinetics of impurity-surface interactions, including adsorption (deposition) and desorption (reentrainment). (Air Liquide)


A new apparatus that enables sampling particles from high-pressure electronic specialty gases is described. The apparatus consists of a patented flow control unit and a commercial particle sensor. The new technique is used to sample particles from several gases. (Air Liquide)


This paper discusses the purity performance and operating characteristics of a Point of Use chemical generator. Ammonium Hydroxide and Hydrochloric Acid were chosen for study because they are widely used in wafer cleaning applications. ICP-MS and GFAA were performed on the solutions. (SEMATECH, AT&T Bell Labs)


This paper presents case histories of practical applications of APIMS measurement technology in the SEMATECH UHP distribution system. Bulk argon supply measurements were made in conjunction with replacement of process tools; locations included prior to argon purifier, immediately downstream of the purifier, and at a distant service point. The APIMS was used to provide a baseline characterization of a new bulk nitrogen supply system. (SEMATECH, Praxair, Inc.)


Willis, C., "Liquid Particle Counter Comparison," In *Particles in Gases and Liquids 1: Detection, Characterization, and Control*, ed. K. L. Mittal. New York: Plenum Press, 1989, pp. 81-96. This paper discusses liquid laser particle counters from two manufacturers and compares them by the simultaneous measurement of particles in deionized (DI) water and in various acids. Good correlation was observed for certain size ranges; for others, the numbers were dissimilar but predictable. The particle distribution was characteristic for each counter type, and consistent for the different fluids examined. (Texas Instruments)


Wu, Y., T. H. Kuehn, and B. Y. H. Liu, "Simulation of Flows and Contaminant Transport in Liquid Tanks," In Proceedings of the 39th Annual Technical Meeting of the Institute of Environmental Sciences, 1993, pp. 396-406. This paper discusses the development of a simulation technique that can be used to predict the flow due to combined pumping, natural convection and acoustic streaming. The transport of both chemicals and particulate contaminants is also discussed. (University of Minnesota)


Yagi, Y., F. Hayashi, and Y. Uchitomi, "Evaluation of Boron Behavior in Ultrapure Water Manufacturing System," In Annual Semiconductor Pure Water and Chemicals Conference Proceedings, 1994, pp. 54-63. This paper discusses the behavior of boron in an ultrapure water system. The adherence of boron on the Si wafer surface was also studied. ICP-MS analysis method and SFP method were used to determine boron concentration in water. Boron concentration was found at 50 to 60 ppb in city water and 0.1 ppb or less in the ultrapure water manufacturing system. Removal of boron is achieved by means of anion exchange resin. Boron contamination is deemed improbably since it is found in ultrapure water at less than 0.1 ppb. (Hitachi Co.)


The paper discusses the Nomura Microscience Method used to determine differences between two ultrapure water distribution systems. The total system (including monitoring points) approach is essential for obtaining stable and high quality ultrapure water. A data collection and management system is necessary as more and more on-line monitoring instruments are installed in the ultrapure water system. Data analysis and correlation studies can then be retrieved accurately and rapidly. The performance of state of the art micro or ultra filters can only be revealed by using particle counting methods with better than 0.1 micron sensitivity. (Motorola)

This paper discusses modifications to the technology of an oscillating crystal hygrometer for reliable use under 10 ppb moisture in inerts, oxygen, and hydrogen. This performance has been validated by testing with certified moisture standards. (Air Products and Chemicals, Inc., Ametek)

This paper presents data from a moisture certification laboratory coordinated with analytical technology research. (Air Products and Chemicals, Inc.)

This paper discusses a procedure to filter particles with diameters as low as 0.2 μm, and statistically quantify them using a scanning electron microscope (SEM) was developed and found to be limited with liquid reagents. Counting particles with this procedure indicated that large differences existed between automated counters and the SEM techniques. (IBM)

(Air Products and Chemicals)