

# Wet Chemical Analysis for the Semiconductor Industry

## A Total View

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The analysis of liquids to obtain information about semiconductor materials is known in the industry as "wet chemistry" and has been used since the beginning of the production of IC's. However, the analytical procedures never gained any significant attention until the mid 70's when the absolute measurement of phosphorus in PSG films by wet chemical analysis was incorporated by several industrial labs as the standard method of analysis.

Today, over 120 different procedures are used to gain specific information about incoming and processed materials used in the industry. These procedures cover ultra pure water, chemicals, thin films, and wafer cleanliness. Furthermore, they are used to evaluate the cleanliness of reactors, cleanrooms, and components of all kinds that are used in cleanrooms, wet benches and reactors.

This paper will cover a total look at the applications of wet chemical processes and the usefulness of the data obtained from these analytical techniques. The paper will cover not only those tests that one would expect to be done by wet processes such as the analysis of metals in chemicals, but will also cover many unusual applications of wet chemical analysis such as their usefulness in evaluating products from a variety of reactors.

Included in this part of the presentation will be a unique application to determine ion implantation contaminants and recent advances for analyzing 300mm wafers without breaking them and the analysis of contamination metals in copper thin films. Actual data will be provided for each of the analytical techniques presented.

### INTRODUCTION

The analysis of liquids and solutions to obtain information about semiconductor materials, processes and the environment in which integrated circuits are produced is known in the semiconductor industry as wet chemistry. Even though wet methods have been used in the industry since the late 50's, there is still little understanding about these techniques. Yet, well over 120 procedures are used to obtain specific information in the industry's effort to maintain good quality control, improve yields and to help understand the sources of contamination.

It is easier to accept that wet analytical procedures can be used to measure materials used on the wet side of manufacturing such as ultra pure water, chemicals, and wet benches. However, as will be shown, wet procedures are also very useful in analyzing processed wafers, thin films, reactors, cleanrooms and components of all kinds.

There are many advantages to using wet chemical methods for measurements when low level specific information is needed for process evaluation and process control such as:

- Wet chemical measurements are primary, easily traceable to standards (i.e., NIST) and therefore absolutely quantitative.
- One can duplicate a matrix exactly and thus determine when and how the matrix causes interference and thus false data.
- Recovery studies can easily be done to verify the method.
- Since the procedures give accurate results, it is possible to compare data over time and to see baseline shifts or variations in processing.
- Wet chemical analysis is extraordinarily sensitive for

measuring materials in the range of percentages down to parts-per-trillion (ppt) or atoms per square centimeter.

These facts not only make wet methods excellent for metrology, but also make them useful to standardize other methods of analysis such as Fourier Transform Infrared (FTIR) or complement "dry" instruments such as TXRF or SIMS.

The most obvious applications for wet chemical analysis are those where the samples are already in a liquid state. In fact for materials in this state, wet analysis is not only the right choice but generally the only choice. For example, tools like inductively coupled plasma-mass spectrometry (ICP-MS) are used to determine the concentration of metals in UPW (see **Table 1**). Furthermore, since water can easily be concentrated, the detection levels shown, in **Table 1**, can be lowered by a factor of 10 or 100. Therefore, parts-per-quadrillion (ppq) levels of metals in UPW can be measured today.

ICP-MS is also useful for measuring metals in chemicals. With chemicals, however, the analyst needs to do some preparation of the sample to be analyzed. Since the major constituent interferes with the accurate measurement of very low concentration of metals, it must be removed. Furthermore, during the preparation step, the sample must not become contaminated. To insure both accurate measurement and that no contamination has occurred, recovery studies are done. **Table 2** shows recoveries for HF after the removal of the matrix. The detection levels in this case are obtainable without concentrating the sample. In other words, after matrix removal the sample is brought back to the original volume.

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**TABLE 1. Trace Metals Detection Limits in UPW by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS).**

| Element           | DL (ppb) |
|-------------------|----------|
| Aluminum (Al)     | 0.003    |
| Antimony (Sb)     | 0.002    |
| Arsenic (As)      | 0.005    |
| Barium (Ba)       | 0.001    |
| Beryllium (Be)    | 0.003    |
| Bismuth (Bi)      | 0.001    |
| Boron (B)         | 0.05     |
| Cadmium (Cd)      | 0.003    |
| Calcium (Ca)      | 0.2      |
| Cerium (Ce)       | 0.001    |
| Cesium (Cs)       | 0.001    |
| Chromium (Cr)     | 0.004    |
| Cobalt (Co)       | 0.001    |
| Copper (Cu)       | 0.003    |
| Dysprosium (Dy)   | 0.001    |
| Erbium (Er)       | 0.001    |
| Europium (Eu)     | 0.001    |
| Gadolinium (Gd)   | 0.001    |
| Gallium (Ga)      | 0.001    |
| Germanium (Ge)    | 0.002    |
| Gold (Au)         | 0.003    |
| Hafnium (Hf)      | 0.006    |
| Holmium (Ho)      | 0.001    |
| Indium (In)       | 0.001    |
| Iridium (Ir)      | 0.002    |
| Iron (Fe)         | 0.02     |
| Lanthanum (La)    | 0.001    |
| Lead (Pb)         | 0.003    |
| Lithium (Li)      | 0.002    |
| Lutetium (Lu)     | 0.001    |
| Magnesium (Mg)    | 0.002    |
| Manganese (Mn)    | 0.002    |
| Mercury (Hg)      | 0.02     |
| Molybdenum (Mo)   | 0.004    |
| Neodymium (Nd)    | 0.001    |
| Nickel (Ni)       | 0.004    |
| Niobium (Nb)      | 0.001    |
| Osmium (Os)       | 0.002    |
| Palladium (Pd)    | 0.002    |
| Platinum (Pt)     | 0.009    |
| Potassium (K)     | 0.1      |
| Praseodymium (Pr) | 0.001    |
| Rhenium (Re)      | 0.003    |
| Rhodium (Rh)      | 0.001    |
| Rubidium (Rb)     | 0.001    |
| Ruthenium (Ru)    | 0.002    |
| Samarium (Sm)     | 0.002    |
| Scandium (Sc)     | 0.01     |
| Selenium (Se)     | 0.02     |
| Silicon (Si)      | 0.5      |
| Silver (Ag)       | 0.001    |
| Sodium (Na)       | 0.007    |
| Strontium (Sr)    | 0.001    |
| Tantalum (Ta)     | 0.004    |
| Tellurium (Te)    | 0.005    |
| Terbium (Tb)      | 0.001    |
| Thallium (Tl)     | 0.006    |
| Thorium (Th)      | 0.003    |

|                |       |
|----------------|-------|
| Thulium (Tm)   | 0.001 |
| Tin (Sn)       | 0.005 |
| Titanium (Ti)  | 0.002 |
| Tungsten (W)   | 0.005 |
| Uranium (U)    | 0.002 |
| Vanadium (V)   | 0.003 |
| Ytterbium (Yb) | 0.001 |
| Yttrium (Y)    | 0.001 |
| Zinc (Zn)      | 0.005 |
| Zirconium (Zr) | 0.005 |

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**TABLE 2. Detection Limits and Recoveries in 49% HF by ICP-MS.**

| Element         | DL ppb | % Recovery |
|-----------------|--------|------------|
| Aluminum (Al)   | 0.01   | 114        |
| Antimony (Sb)   | 0.01   | 99         |
| Barium (Ba)     | 0.005  | 109        |
| Beryllium (Be)  | 0.01   | 102        |
| Boron (B)       | 0.2    | 82         |
| Cadmium (Cd)    | 0.01   | 90         |
| Calcium (Ca)    | 0.3    | 118        |
| Chromium (Cr)   | 0.01   | 89         |
| Cobalt (Co)     | 0.005  | 88         |
| Copper (Cu)     | 0.01   | 85         |
| Gallium (Ga)    | 0.005  | 94         |
| Germanium (Ge)  | 0.01   | 90         |
| Iron (Fe)       | 0.3    | 91         |
| Lead (Pb)       | 0.01   | 114        |
| Lithium (Li)    | 0.005  | 104        |
| Magnesium (Mg)  | 0.01   | 117        |
| Manganese (Mn)  | 0.01   | 94         |
| Molybdenum (Mo) | 0.01   | 91         |
| Nickel (Ni)     | 0.01   | 91         |
| Niobium (Nb)    | 0.05   | 93         |
| Potassium (K)   | 0.3    | 112        |
| Silver (Ag)     | 0.01   | 107        |
| Sodium (Na)     | 0.01   | 98         |
| Strontium (Sr)  | 0.005  | 105        |
| Tantalum (Ta)   | 0.05   | 84         |
| Thallium (Tl)   | 0.05   | 97         |
| Tin (Sn)        | 0.01   | 96         |
| Vanadium (V)    | 0.01   | 88         |
| Zinc (Zn)       | 0.05   | 80         |
| Zirconium (Zr)  | 0.01   | 99         |

By using a variety of sample preparation procedures, chemicals of all types can be analyzed for metal content. **Table 3** contains a list of chemicals used in the semiconductor industry that are routinely analyzed. Furthermore, these chemicals can be measured to determine other aspects of their composition, such as concentration, anions and cations, organic contamination, (TOC in aqueous chemicals or specific compounds in organic solvents), moisture in organic materials and other specific tests to characterize the chemicals.

**TABLE 3.** List Of Chemicals Analyzed By Wet Chemical Procedures

| Chemical                 |                            |
|--------------------------|----------------------------|
| 1-Methyl-2-Pyrrolidone   | 2-Propanol                 |
| Acetic Acid              | Acetone                    |
| Ammonium Hydroxide       | Hexamethyldisilazane       |
| Hydrochloric Acid        | Hydrofluoric Acid          |
| Hydrogen Peroxide        | Methanol                   |
| Methyl Ethyl Ketone      | Methyl Isobutyl Ketone     |
| Mixed Acid Etchants      | n-Butyl Acetate            |
| Nitric Acid              | SC1 Cleaning Solution      |
| SC2 Cleaning Solution    | Sulfuric Acid              |
| TMAH                     | Ammonium Fluoride Solution |
| Buffered Oxide Etchants  | Resist Strippers           |
| Negative Photoresists    | Positive Photoresists      |
| Polyimide Solutions      | Polyimide Resins           |
| Photosensitizers         | Photoresist Resins         |
| Ethylene Glycol          | Polymeric Materials        |
| Polypropylene            | Polyethylene               |
| Polyfluorocarbons        | Epoxy Resins               |
| Non-Ionic Surfactants    | Spin-on-Glass              |
| Spin-on-Boron            | Spin-on-Phosphorus         |
| Tetraethyl Orthosilicate | Phosphorus Oxychloride     |
| Germanium Tetrachloride  | Trimethylphosphite         |

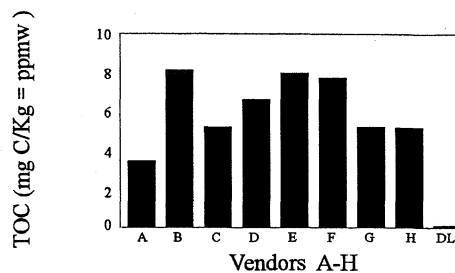
Of these, TOC in etching and cleaning chemicals is frequently overlooked as a contaminant that could be harmful in cleaning chemicals. Generally they have not been observed to cause serious problems. However, considering that organic contaminants at ppmw levels are usually found in chemicals, they may be more deleterious in future processing (see Table 4). Figures 1a and 1b show the content of organic contaminants in hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>). Figure 2 shows the variation in concentration of TOC in sulfuric acid by vendor, lot and container type. Although these materials are oxidized under extreme conditions they could still cause problems under normal use, for example forming SiC on wafers.

**TABLE 4.** Summary of Current Detection Limits and Analytical Spike Recoveries for TOC in Selected Chemicals.

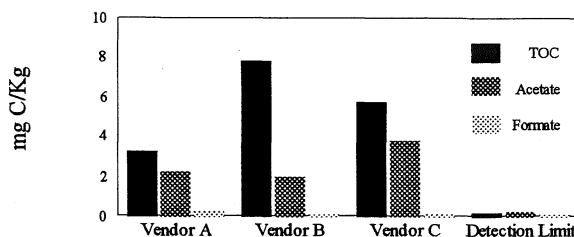
| Chemical                    | Average Recovery** | TOC Detection Limits ppmw |
|-----------------------------|--------------------|---------------------------|
| 50-100% Sulfuric Acid       | 93%                | 0.2*                      |
| 85% Phosphoric Acid         | 97%                | 0.1*                      |
| 25-51% Hydrofluoric Acid    | 97%                | 0.1*                      |
| 35-71% Nitric Acid          | 99%                | 0.5*                      |
| 1-40% Hydrogen Peroxide     | 103%               | 0.1                       |
| Ammonium Hydroxide          |                    |                           |
| 28-31% NH <sub>3</sub>      | 99%                | 0.2*                      |
| 50% Sodium Hydroxide        | 96%                | 0.4*                      |
| 45% Potassium Hydroxide     | 102%               | 0.4*                      |
| 40% Ammonium Fluoride       | 98%                | 0.1                       |
| BOE, Buffered Oxide Etchant | 99%                | 0.1 - 0.2*                |

\* Lower detection limits may be possible for more dilute solutions.

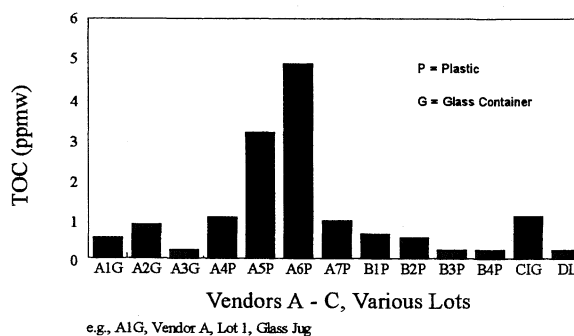
\*\* Spike recoveries are for an organic standard (morpholine, C<sub>4</sub>H<sub>9</sub>NO) added after sample preparation. Calibration was performed using the same standard in deionized water.



**FIGURE 1a.** TOC in 30% Hydrogen Peroxide.



**FIGURE 1b.** TOC, Acetate And Formate Found In H<sub>2</sub>O<sub>2</sub> After Catalytic Decomposition.



**FIGURE 2.** TOC in Concentrated H<sub>2</sub>SO<sub>4</sub>.

The most serious aspect in analyzing UPW and chemicals is preventing contamination while preparing the sample for measurement particular in the case of metals at the ppb, ppt and now sub-ppt levels. To reach the low levels sample collecting, vessel cleaning and concentration become extraordinarily important. While cleanroom conditions are adequate for sample collecting and vessel cleaning, they are not for sample concentration or measurement. Special equipment such as closed evaporators and automatic sampling units for ion coupled plasma-mass spectrometer (ICP-MS) or graphite furnace atomic absorption spectrometer (GFAAS) where samples are in a holding pattern require filtered N<sub>2</sub> environments (see Figure 3). Although the final measurement can be a ten minute step, the extraordinary activity to preparing samples for measurement in absolutely clean and safe environments can take hours.

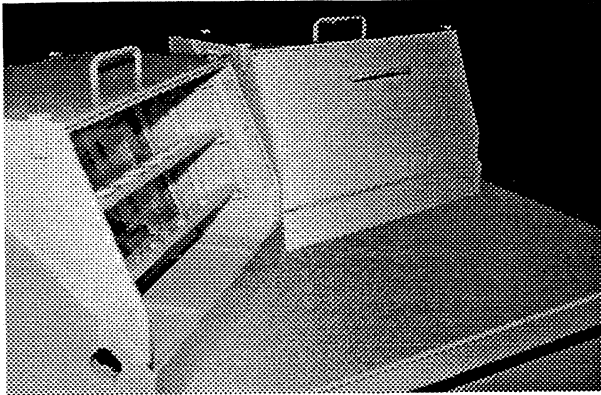


FIGURE 3. Closed Evaporator

Another important wet chemical application for the semiconductor industry is the measurement of metals on bare wafers, or in thin and thick oxides, nitrides or metallic films such as copper. To verify the accuracy of this technique, known quantities of metals from NIST standards are deposited directly on wafers declared clean by having gone through a Vapor Phase Decomposition - Inductively Coupled Plasma-Mass Spectrometry (VPD-ICP-MS) analysis just prior to being used. The solution is placed over the entire wafer, dried in a protective environment and processed (see Table 5).

In Table 5, it becomes very clear why recovery studies are so important. As noted, copper did not come off of the wafer using the standard VPD process. To remove copper efficiently another process known as drop scan ICP-MS is used. In this process any mixture of acids, bases, oxidizers or chelating materials can be made to remove specific metals from wafers. Using this procedure, copper can be removed efficiently, with 95% recovery.

Having methods to accurately measure metals in chemicals and in chemical baths, a correlation can now be made between metals in chemicals or UPW and those found to adhere to wafers (see Table 6). In this table two grades of hydrogen peroxide were evaluated after one proved to cause yield loss while the other did not. Although both chemicals were pure, especially considering the process where they were being used, the enhanced metals plating onto wafers in SC1 created a yield loss situation even though an SC2 clean followed. It has been observed through other failures involving metals on wafers that iron quantities over  $15 \times 10^{10}$  atoms/cm<sup>2</sup> generally cause failures.

Moving away from liquid and wafers into other areas, one where wet chemical analysis has proved to be extraordinarily useful is the measurement of cleanroom molecular contamination. A serious problem in production is hazy wafers. Although there are a wide variety of reasons for hazy wafers to occur such as particles, roughness, adsorbed organics or metal oxides such as Fe<sub>2</sub>O<sub>3</sub>, one cause is salt formations. These salts deposit on wafers directly when vapors such as HCl, HF or SO<sub>3</sub> react with NH<sub>3</sub> or organic amines. By scrubbing air through UPW or dilute chemical solutions, these contaminants known as "molecular contaminants" can be measured. Whether metallic, processing fumes, outgassed or leachable materials, these agents can be identified and usually related to both their source and effect on the wafer.

TABLE 5. Recovery Study of Selected Elements by Vapor Phase Decomposition - Inductively Coupled Plasma Mass Spectrometry (VPD-ICP-MS).

| Wafer No.            | Spike [ng] | Recovered [ng] | Blank [ng] | Percent Recovery |
|----------------------|------------|----------------|------------|------------------|
| <b>Aluminum</b>      |            |                |            |                  |
| 1                    | 1.26       | 1.02           | 0.076      | 89               |
| 2                    | 2.35       | 2.30           | 0.421      | 90               |
| 3                    | 4.47       | 4.36           | 0.038      | 94               |
|                      |            |                | AV = 0.178 |                  |
| <b>Chromium</b>      |            |                |            |                  |
| 1                    | 0.654      | 0.644          | <0.02      | 99               |
| 2                    | 1.22       | 1.10           | <0.02      | 90               |
| 3                    | 2.32       | 2.28           | <0.02      | 98               |
|                      |            |                | AV = 0.02  |                  |
| <b>Copper (*)</b>    |            |                |            |                  |
| 1                    | 0.541      | 0.024          | <0.01      | 4.4              |
| 2                    | 1.01       | <0.02          | <0.01      | <2.0             |
| 3                    | 1.92       | 0.159          | <0.01      | 8.3              |
|                      |            |                | AV = 0.01  |                  |
| <b>Nickel</b>        |            |                |            |                  |
| 1                    | 0.567      | 0.607          | <0.01      | 107              |
| 2                    | 1.06       | 0.978          | 0.02       | 92               |
| 3                    | 2.01       | 2.01           | <0.01      | 100              |
|                      |            |                | AV = 0.01  |                  |
| <b>Iron by GFAAS</b> |            |                |            |                  |
| 1                    | 1.08       | 1.20           | -          | 103              |
| 2                    | 2.21       | 2.58           | 0.06       | 113              |
| 3                    | 2.95       | 3.29           | 0.11       | 109              |
|                      |            |                | AV = 0.085 |                  |
| <b>Sodium</b>        |            |                |            |                  |
| 1                    | 1.79       | 2.12           | 0.190      | 87               |
| 2                    | 3.34       | 3.79           | 0.857      | 96               |
| 3                    | 6.35       | 6.77           | 0.660      | 98               |
|                      |            |                | AV = 0.569 |                  |

Remark (\*): Another drop scanning method is used to get quantitative recovery for copper.

TABLE 6. Comparison of Trace Metal Concentration in Hydrogen Peroxide Solutions Used in SC1 Clean and on 6" bare Wafers

| Element       | Concentration (ppb)<br>Hydrogen Peroxide |         | Surface Concentration<br>( $\times 10^{10}$ atoms/cm <sup>2</sup> ) |         |
|---------------|--|---------|---|---------|
|               | Grade A                                  | Grade B | Wafer A   | Wafer B |
| Aluminum (Al) | 22                                       | 2.6     | 580   | 92      |
| Chromium (Cr) | 3.3                                      | 0.1     | <1  | <1      |
| Iron (Fe)     | 6.7                                      | <2      | 82  | <5      |
| Nickel (Ni)   | 2.6                                      | <0.1    | 1.2   | 0.9     |
| Sodium (Na)   | 10                                       | 2.3     | 12  | 14      |
| Total         | 45                                       | 7       | >600  | >100    |

A company with just such a haze problem solicited help to find its cause and remove it. A wafer was evaluated by leaching it with UPW and running ion chromatography (IC) where results like those shown in Table 7 are given. A wafer was also evaluated by VPD-ICP-MS. Finding that salt formation was the cause of the haze, air samples were run. Significant quantities of acid and base fumes were detected but no significant amount of metals.

**TABLE 7. Surface concentration of Anions and Cations on 150mm Wafers by IC. (surface area: 364 cm<sup>2</sup>, volume: 200 mL)**

| Ion            | DL<br>ions/cm <sup>2</sup> | A<br>ions/cm <sup>2</sup> | B<br>ions/cm <sup>2</sup> | C<br>ions/cm <sup>2</sup> | Typical Range<br>ions/cm <sup>2</sup> |
|----------------|----------------------------|---------------------------|---------------------------|---------------------------|---------------------------------------|
| <b>Anions</b>  |                            |                           |                           |                           |                                       |
| Fluoride       | 1.7 E+12                   | *                         | *                         | *                         | <DL - 5 E+13                          |
| Chloride       | 1.9 E+11                   | 1.7 E+12                  | 2.0 E+12                  | 3.3 E+12                  | <DL - 5 E+13                          |
| Nitrite        | 1.4 E+11                   | 1.1 E+12                  | 9.3 E+11                  | 1.5 E+12                  | <DL - 1 E+13                          |
| Bromide        | 8.3 E+11                   | *                         | *                         | *                         | <DL                                   |
| Nitrate        | 1.1 E+11                   | 5.9 E+11                  | 1.1 E+12                  | 1.4 E+12                  | <DL - 1 E+13                          |
| Phosphate      | 7.0 E+11                   | *                         | *                         | *                         | <DL                                   |
| Sulfate        | 1.7 E+11                   | 4.8 E+11                  | 1.8 E+12                  | 7.9 E+11                  | <DL - 2 E+13                          |
| <b>Cations</b> |                            |                           |                           |                           |                                       |
| Lithium        | 4.8 E+11                   | *                         | *                         | *                         | <DL                                   |
| Sodium         | 1.4 E+11                   | 1.9 E+12                  | 1.3 E+13                  | 3.2 E+12                  | <DL - 1 E+13                          |
| Ammonium       | 9.2 E+11                   | 5.3 E+12                  | 8.3 E+12                  | 1.0 E+13                  | 1E+12 - 2E+13                         |
| Potassium      | 1.7 E+11                   | *                         | *                         | 1.2 E+12                  | <DL - 4 E+12                          |
| Magnesium      | 2.7 E+12                   | *                         | *                         | *                         | <DL - 2 E+12                          |
| Calcium        | 1.6 E+11                   | *                         | *                         | 1.6 E+12                  | <DL - 4E+12                           |

Changes in air flowrate, fresh air addition to cleanroom air and ventilation of the cleaning stations were made while air sampling continued. Table 8 shows the improvement and the concentration of fumes finally obtained (2/94 → 5/94). These concentrations completely allowed for processing without haze formation. However, it must be stated, not seeing salts as haze on wafers does not mean that none are there. In routine evaluation of anions and cations on wafers after cleaning, we find small quantities of Cl<sup>-</sup>, SO<sub>4</sub><sup>=</sup> and NO<sub>x</sub><sup>-</sup>. The typical ranges that have been measured in a cleanroom of class 10 or better and regardless of the size of the IC manufacturer are shown in Table 9. Amine detection and identification are shown in Figure 4. Figure 5 shows the Balazs Analytical Laboratory Air Sampler used in this study.

Another significant wet chemical application has been the measurement of metals in thin and thick films. A study of oxide films is shown in Table 10. Thermal, deposited and TEOS wafers were analyzed from several different sources. As is seen, the concentrations can be significant. Even though the situation has improved, metals in oxides, nitrides and other films are still quite high in films from various reactors today.

**TABLE 8. Non-metallic Contamination at Various Sites in A Class 100 Cleanroom.**

| Element<br>ug/m <sup>3</sup> | Class 100 Cleanroom |       |            |       |            |       |
|------------------------------|---------------------|-------|------------|-------|------------|-------|
|                              | Location 1          |       | Location 2 |       | Location 3 |       |
|                              | 2/94                | 5/94  | 2/94       | 5/94  | 2/94       | 5/94  |
| Ammonium                     | 61                  | 7.9   | 12         | 4.4   | 3.0        | 2.9   |
| Fluoride                     | 28                  | 1.4   | 10         | 1.1   | 2.9        | 1.6   |
| Chloride                     | 0.14                | <0.01 | 0.045      | <0.01 | 0.14       | 0.16  |
| Nitrite                      | 33                  | 3.3   | 9.0        | 1.4   | 6.9        | 3.7   |
| Phosphate                    | <0.02               | <0.02 | <0.02      | <0.02 | <0.02      | <0.02 |
| Bromide                      | <0.02               | <0.02 | <0.02      | <0.02 | <0.02      | <0.02 |
| Nitrate                      | 3.1                 | 0.67  | 0.94       | 0.40  | 1.6        | 0.82  |
| Sulfate                      | 0.14                | 0.04  | 0.16       | <0.02 | 0.51       | 0.25  |

**TABLE 9. Cleanroom Air Analysis: Acids and Bases. Analysis using water scrubbers then IC.**

| Anhydrides: |                                  | Typical Range        |                |
|-------------|----------------------------------|----------------------|----------------|
|             |                                  | (ng/m <sup>3</sup> ) | pptv*          |
| Ammonium    | (NH <sub>4</sub> <sup>+</sup> )  | 1,000 - 10,000       | 1,400 - 14,000 |
| Bromide     | (Br <sup>-</sup> )               | <30                  | <9             |
| Chloride    | (Cl <sup>-</sup> )               | 100-1,500            | 70 - 1,000     |
| Fluoride    | (F <sup>-</sup> )                | <600                 | <800           |
| Nitrate     | (NO <sub>3</sub> <sup>-</sup> )  | 500 - 5,000          | 200 - 2,000    |
| Nitrite     | (NO <sub>2</sub> <sup>-</sup> )  | 1,000 - 10,000       | 530 - 5,300    |
| Phosphate   | (HPO <sub>4</sub> <sup>=</sup> ) | <20                  | <5             |
| Sulfate     | (SO <sub>4</sub> <sup>=</sup> )  | <50 - 500            | <13 - 130      |

\* pptv = parts-per-trillion-volume

**TABLE 10. Typical Concentration of Trace Metals In Thermal, TEOS and Grown or Deposited Oxide Layers From Different Sources. Surface Concentration in 10<sup>10</sup> atoms/cm<sup>2</sup>.**

| Sources   |      | Thermal |     |      |      | TEOS Deposited |      |     |      |      |
|-----------|------|---------|-----|------|------|----------------|------|-----|------|------|
|           |      | A       | B   | C    | D    | E              | F    | G   | H    | I    |
| Aluminum  | (Al) | 440     | 180 | 2400 | 5400 | 20             | 3000 | 95  | 70   | <25  |
| Sodium    | (Na) | -       | -   | -    | -    | <50            | 450  | <30 | <20  | <20  |
| Chromium  | (Cr) | 94      | 42  | 550  | 100  | <1             | -    | <2  | <0.7 | <0.7 |
| Iron      | (Fe) | 340     | 330 | 900  | 440  | <10            | 150  | 40  | <7   | <7   |
| Nickel    | (Ni) | 160     | 110 | 320  | 80   | <1             | -    | <1  | <0.7 | 1.4  |
| Copper    | (Cu) | 160     | 150 | 20   | -    | -              | -    | -   | -    | -    |
| Magnesium | (Mg) | -       | -   | -    | -    | -              | 30   | -   | -    | -    |
| Zinc      | (Zn) | -       | -   | -    | -    | -              | 30   | <5  | <3   | <3   |
| Zirconium | (Zr) | -       | -   | -    | -    | -              | 2.5  | -   | -    | -    |

1991 Study

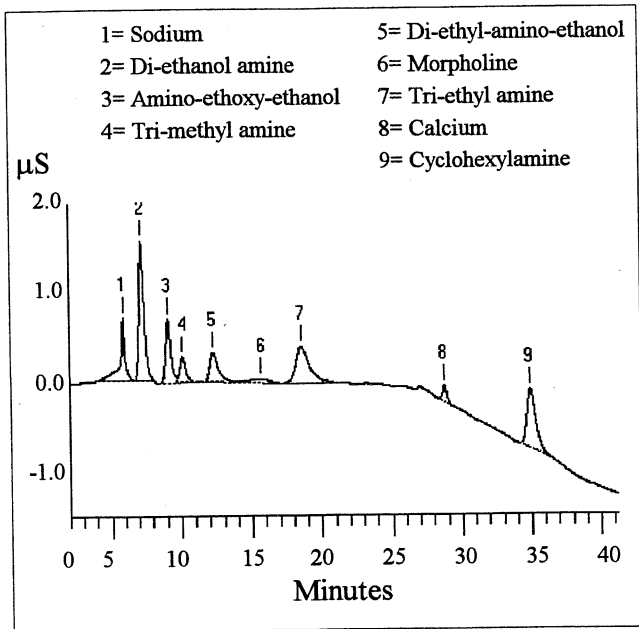


FIGURE 4. Amine Detection by IC.

Chemicals, wet stations, reactors and air are not the only sources of ionic and metallic contamination in a cleanroom. Components of all kinds contain these contaminants which leach out into the air or directly onto wafers. Table 11 lists both construction materials and components measured. To date, over 300 materials have been studied that go into cleanrooms, wet benches, reactors and construction materials of all kinds for UPW, chemical distribution, cleanroom construction, reactors and processing components. Numerous suppliers have requested studies for ions, metal, TOC, particle, deterioration or stability of materials and components in both liquid and vapor atmospheres. Table 12 shows typical leachable ionic contaminants from cleanroom wipes and garments. Table 13 shows the quantity of anions found when three different wafer carriers were rinsed with UPW.

TABLE 11. Materials

|             |                                  |
|-------------|----------------------------------|
| Wafers      | Paints                           |
| O-Rings     | Floor Tiles                      |
| Gloves      | Tubing                           |
| Finger Cots | Potting Compounds                |
| Metals      | Motor Parts                      |
| Filters     | Pumps                            |
| Gaskets     | Safety Glasses                   |
| Hats        | Polymers of all Kinds            |
| Plastics    | Carriers                         |
| Paper       | Wafer Boxes                      |
| Valves      | Cassettes                        |
| Pipes       | Packing Materials                |
| Garments    | Photoresist                      |
| Resins      | Cleanroom Construction Materials |

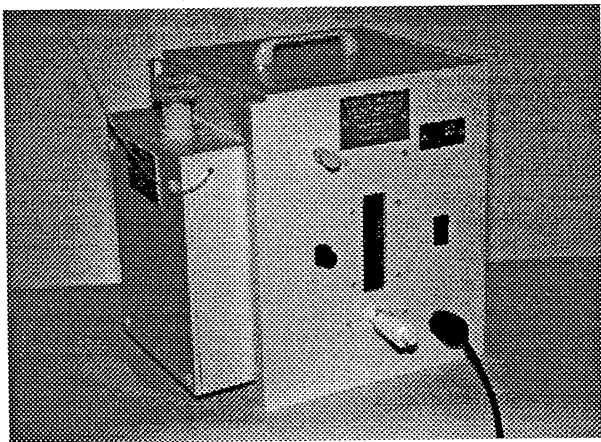


FIGURE 5. Balazs Analytical Laboratory Air Sampler

TABLE 12. Extractable Ionics from Cleanroom Components

| Ion            | Cleanroom Wipes |                    | Cleanroom Garments |                    |
|----------------|-----------------|--------------------|--------------------|--------------------|
|                | DL              | ng/cm <sup>2</sup> | DL                 | ng/cm <sup>2</sup> |
| <b>Anions</b>  |                 |                    |                    |                    |
| Fluoride       | 0.6             | *                  | 10.                | *                  |
| Chloride       | 0.07            | 3.9                | 1.3                | 315                |
| Nitrite        | 0.07            | 0.10               | 1.3                | 2                  |
| Phosphate      | 0.15            | *                  | 2.5                | *                  |
| Bromide        | 0.15            | *                  | 2.5                | 8                  |
| Nitrate        | 0.15            | 0.77               | 2.5                | 2                  |
| Sulfate        | 0.15            | 0.54               | 2.5                | 1600               |
| <b>Cations</b> |                 |                    |                    |                    |
| Lithium        | 0.07            | *                  | 1.3                | *                  |
| Sodium         | 0.07            | 1.7                | 1.3                | 844                |
| Ammonium       | 0.15            | 2.4                | 2.5                | *                  |
| Potassium      | 0.15            | 2.3                | 2.5                | 235                |
| Magnesium      | 0.15            | 0.65               | 2.5                | 224                |
| Calcium        | 0.07            | 2.4                | 1.3                | 2786               |

**TABLE 13.** Surface Concentration of Anions on Wafer Carrier by IC (surface area: 3600 cm<sup>2</sup>, volume: 500 mL, leaching temp.: ambient)

| Ion           | DL<br>ions/cm <sup>2</sup> | A<br>ions/cm <sup>2</sup> | B<br>ions/cm <sup>2</sup> | C<br>ions/cm <sup>2</sup> | Typical Range<br>ions/cm <sup>2</sup> |
|---------------|----------------------------|---------------------------|---------------------------|---------------------------|---------------------------------------|
| <b>Anions</b> |                            |                           |                           |                           |                                       |
| Fluoride      | 8.8 E+12                   | *                         | *                         | *                         | <DL                                   |
| Chloride      | 4.7 E+10                   | 6.1 E+11                  | 4.0 E+11                  | 4.5 E+11                  | <DL - 6 E+11                          |
| Nitrite       | 3.6 E+10                   | 4.0 E+11                  | 2.9 E+11                  | 2.4 E+11                  | <DL - 5 E+11                          |
| Bromide       | 2.1 E+10                   | *                         | *                         | *                         | <DL                                   |
| Nitrate       | 2.7 E+10                   | 1.3 E+11                  | 1.2 E+11                  | 8.1 E+12                  | <DL - 3 E+11                          |
| Phosphate     | 1.8 E+10                   | *                         | *                         | *                         | <DL                                   |
| Sulfate       | 4.4 E+10                   | 1.1 E+11                  | 6.1 E+10                  | *                         | <DL - 3 E+11                          |

Contaminants that can cause n or p type doping are becoming an increasing problem in cleanrooms and on wafers. These materials can come from low level borates in UPW. However, UPW has generally not been the source of dopants that cause yield problems. The source is generally from the air or wafer containers. Wet chemical processes often can measure the boron (B) and phosphorus (P) but their sources are usually identified by using organic adsorbers and GC-MS analytical measuring procedures, because the dopants generally came from organic materials. **Tables 14 and 15** show the quantities of boron (B) and phosphorus found in various materials and from reactors.

**TABLE 14.** B and P in Various Components and Environments ng/cm<sup>2</sup>.

| Component or Environment  | B    | DL    | P    | DL   |
|---------------------------|------|-------|------|------|
| Garment                   | 0.05 | 0.005 | 0.57 | 0.02 |
|                           | 0.09 |       | 2.0  |      |
| Plastic Container (clear) | 0.39 | 0.02  | 0.34 | 0.02 |
|                           | 0.01 |       |      |      |
| (colored)                 | 19.0 |       |      |      |
| Tape (Adhesive)           | 1.8  | 0.02  | 69.  | 0.03 |
| Gasket (Rubber)           | 0.31 | 0.01  | 51   | 0.4  |
| Wafer Box                 | 35.  |       | 12.  |      |
| Mini Environment          | 0.04 | 0.02  | ND   |      |
| Inspection Room           | 0.06 |       | ND   |      |
| HEPA                      | 0.06 |       | ND   |      |

**TABLE 15.** B and P on Bare Wafers 10<sup>10</sup> atoms/cm<sup>2</sup>

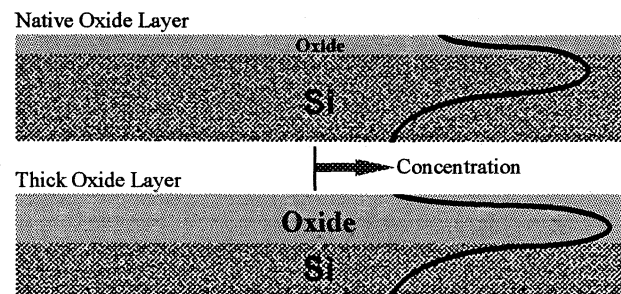
| Ranges on Bare Wafers* | B       | P      |
|------------------------|---------|--------|
|                        | 39-1000 | 12-290 |
| Reactor 1              | 4-90    | 12-19  |
| Reactor 2              | 200-350 | 60-210 |
| Reactor 3              | 20-32   | ND-8   |

\* From different reactors and wafer position in the reactor.

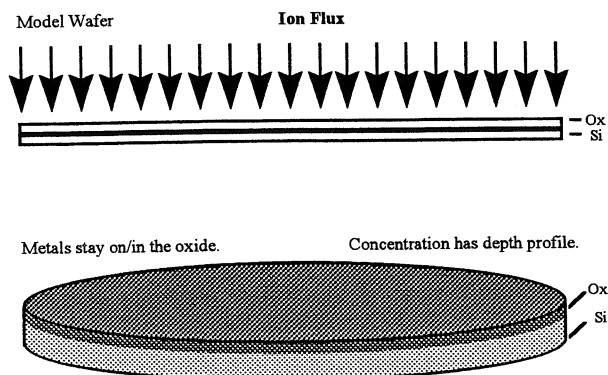
A serious case of n type contamination recently occurred in a fab where someone accidentally hit a water pipe with considerable force causing it to break. Water sprayed in high quantities across the fab. The fab was wiped down using large quantities of cleanroom wipes. After the incident n type doping contamination occurred uncontrollably. A study of air, surfaces, wipes and witness wafers identified the compound (an organophosphate), and its source (the cleanroom wipes). The fab had to be thoroughly wiped down and cleaned and considerable air exchanged before the problem disappeared.

Although not the subject of this paper, it is worth mentioning that the combination of wet chemistry, organics molecular measurements and contaminants found on wafers tied together very well for identifying contaminating materials and their sources. HEPA, ULPA, potting compounds, components of all kinds and wet benches fill the "cleanroom" with huge quantities of contaminants deleterious to wafers and product yield.

A novel application for measuring metals in thick films is to determine those that are implanted into a substrate during the doping process using an ion implanter. Since the different concentration profiles for implanting elements in a substrate during ion implantation is nearly the same as in an oxide film (see **Figure 6**), a wafer with about 2000 Å of oxide on it can become a test wafer. The ion flux implants the dopant and contamination metals into the oxide layer (see **Figure 7**). This layer is then analyzed by drop scan ICP-MS.



**FIGURE 6.** Concentration Profiles After Ion Implantation in Silicon and Silicon Dioxides.



**FIGURE 7.** Measuring Trace Metals on Wafers from Ion Implantation Equipment.

The metals on the surface are removed and analyzed before the metals in the oxide are measured. A control wafer must be run to determine the metals in the oxide film which has not been exposed to the ion implanter. Table 16 reveals a fairly dirty ion implantation process. With this information, however, the engineers were able to greatly improve the situation.

Having measured numerous wafers for metallic contamination from ion implanters, the example above was not very unusual. The range for both surface metallic contamination and implanted metallic contamination is shown in Table 17a and 17b.

**TABLE 16.** High Energy Metallic Contamination (of Oxide) from an Ion Implanter. Trace Metals in Oxide Film on 150 mm Silicon Wafers  $10^{10}$  atoms/cm<sup>2</sup>.

| Element        | Detection Limit | Sample |     |         | Implanted Metal |
|----------------|-----------------|--------|-----|---------|-----------------|
|                |                 | #1     | #2  | Control |                 |
| Calcium (Ca)   | 1.0             | 42     | 39  | 24      | 17              |
| Potassium (K)  | 0.2             | 18     | 12  | 8.5     | 7               |
| Sodium (Na)    | 0.5             | 22     | 14  | 16      | 2               |
| Aluminum (Al)  | 0.5             | 290    | 280 | 18      | 267             |
| Iron (Fe)      | 0.5             | 34     | 35  | 9.0     | 26              |
| Chromium (Cr)  | 0.5             | 4.7    | 3.0 | 0.8     | 3               |
| Nickel (Ni)    | 0.4             | 9.0    | 11  | 3.6     | 7               |
| Zinc (Zn)      | 0.2             | 18     | 17  | 19      | 0               |
| Lithium (Li)   | 0.1             | *      | *   | *       | *               |
| Beryllium (Be) | 0.3             | *      | *   | *       | *               |
| Magnesium (Mg) | 0.6             | 18     | 12  | 5.5     | 14              |
| Titanium (Ti)  | 0.8             | 236    | 45  | *       | 141             |
| Tungsten (W)   | 0.01            | 35     | 27  | *       | 31              |

\* None detected above the detection limit.

**TABLE 17a.** Surface Metallic Contamination After Ion Implantation

| Element  | Concentration Range ( $1 \times 10^{10}$ atoms/cm <sup>2</sup> ) |
|----------|--|
| Aluminum | 100 - 10,000   |
| Iron     | 10 - 100   |
| Chromium | 1 - 10   |
| Nickel   | 1 - 10   |
| Titanium | 10 - 1,000   |

**TABLE 17b.** Range of Metal Contaminants Implanted Into the Substrate.

| Element  | Concentration Range ( $1 \times 10^{10}$ atoms/cm <sup>2</sup> ) |
|----------|--|
| Aluminum | 10 - 10,000  |
| Iron     | 10 - 10,000  |
| Chromium | 10 - 5,000   |
| Nickel   | 10 - 5,000   |
| Titanium | 0.1 - 50   |

The accurate measurement of thin film composition is made easy using wet chemical techniques. The film is simply stripped off of the wafer and the elements of interest measured by colorimetry, ICP-MS, GFAAS or inductively coupled plasma - optical emission spectrometer (ICP-OES). PSG and BPSG are measured using colorimetry or ICP-OES. By getting absolute data traceable to NIST standards, an engineer can standardize secondary equipment such as FTIR or x-ray. It is important to understand, however, that the four methods mentioned (colorimetry, FTIR, ICP-OES, and x-ray) do not measure the same thing and thus often yield different answers. For example ICP-OES and x-ray measure only the total amount of phosphorus while FTIR measures the P-O and P-OH bonds and colorimetry measures only  $PO_4^{3-}$ . By changing the chemistry of the solution, however, colorimetry and only colorimetry can determine the concentration of  $P_2O_5$  vs.  $P_2O_3$  in the oxide film (see Table 18). Also, with colorimetry other compounds of phosphorus have been measured and the discovery made of occluded  $PH_3$  from plasma reactors and the concentrations of this compound measured (see Table 19).

**Table 18.** Concentration on  $P_2O_5$  vs.  $P_2O_3$  in the Oxide Film

| Sample | X-Ray %P | Total P     |          |          |
|--------|----------|-------------|----------|----------|
|        |          | Colorimetry | $P_2O_3$ | $P_2O_5$ |
| 1AD    | 7.6      | 7.5         | 2.7      | 4.8      |
| 2AD    | 5.0      | 4.9         | 1.5      | 3.4      |
| 3AD    | 2.2      | 1.8         | 0.5      | 1.3      |
| 1AN    | 8.0      | 5.9         | 1.1      | 4.8      |
| 2AN    | 5.1      | 4.3         | 0.4      | 3.9      |
| 3AN    | 2.3      | 1.7         | <0.2     | 1.7      |

AD = as deposited, AN = annealed 800 °C in  $N_2$

**TABLE 19.** Weight Percent Phosphorus in Plasma Doped Oxides.

| No. | Total | $P_2O_3$    | $P_2O_5$   | $PH_3$       |
|-----|-------|-------------|------------|--------------|
| A-1 | 9.5   | 6.2 (65.3)* | 2.9 (30.5) | 0.4 (4.2)    |
| A-2 | 5.6   | 4.0 (71.4)  | 1.1 (19.6) | 0.5 (9.0)    |
| A-3 | 5.2   | 3.9 (75.0)  | 0.9 (17.3) | 0.4 (7.7)    |
| A-4 | 5.0   | 4.1 (82.0)  | 0.4 (8.0)  | 0.5 (10.0)   |
| A-5 | 5.4   | 3.9 (72.2)  | 1.0 (18.5) | 0.5 (9.3)    |
| B-1 | 4.1   | 3.3 (80.5)  | 0.6 (14.6) | 0.2 (4.9)    |
| B-2 | 4.1   | 3.4 (82.9)  | 0.5 (12.2) | 0.2 (4.9)    |
| C-1 | 5.0   | 3.8 (76.0)  | 1.1 (22.0) | <0.1 (2.0)   |
| C-2 | 2.3   | 1.9 (82.6)  | 0.4 (17.4) | <0.05 (<1.0) |
| C-3 | 7.5   | 6.2 (82.7)  | 1.3 (17.3) | <0.05 (<1.0) |

\* Numbers in parentheses are relative percents of weight percent phosphorus.



Today with the cost of 300 mm wafers, a change of the wet procedure has made it possible to measure phosphorus and boron in PSG and BPSG and return the intact wafer to the client for re-use. **Tables 20 and 21** compare the old method with the new and is a comparison of data using two completely different techniques. It is this kind of adaptability that illustrates the uniqueness, breadth and value of wet chemical testing.

Other measurements of thin films include Cu or Si in aluminum metalization, titanium/tungsten or silicide ratioing and metals in copper films (see **Table 22**). New methods are continuously being developed to deal with new films being used by the industry.

Studies such as the number of etches required to clean a quartz tool (see **Figure 7**), leachable metals and TOC from FRPP (see **Table 23**), the amount of fluoride contributed to hot UPW or peroxide solutions that go through fluoropolymer tubing, or a dynamic study of 100 foot of new tubing (see **Table 24**), illustrate the diversity of wet chemical analysis.

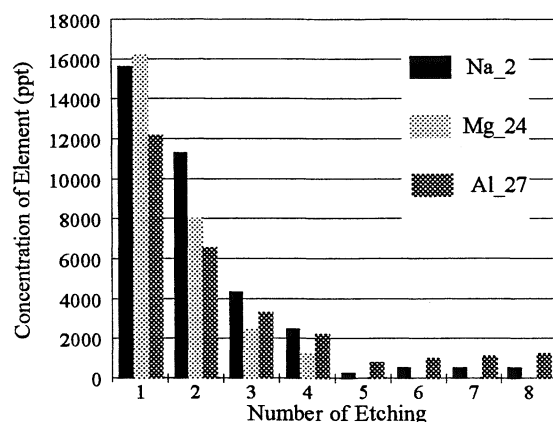
**TABLE 20.** Results for %P and %B in BPSG film on a 300 mm Wafer.

| Method   | %P (wt/wr)  | %B (wt/wt) |
|----------|-------------|------------|
|          | Colorimetry | ICP-OES    |
| Standard | 4.85        | 3.36       |
| New*     | 4.82        | 3.37       |

\* Non-destructive analysis

**TABLE 21.** Non-destructive Analysis of 300 mm Wafers for %P and %B.

| Wafer Sample | Colorimetry | ICP-OES |
|--------------|-------------|---------|
| # 1          | 0.00        | 0.00    |
| # 2          | 1.90        | 2.00    |
| # 3          | 2.68        | 2.69    |
| # 4          | 3.77        | 3.81    |
| # 5          | 4.36        | 4.39    |
| # 6          | 5.72        | 5.71    |
| # 7          | 7.20        | 7.33    |
| # 8          | 8.72        | 8.78    |



**FIGURE 7.** 0.5% HF Etching of Quartz Vessel #3.

**Table 22.** Trace Impurities in Copper Films (ng/g).

| Element         | 8" Wafer | Spike Recovery |
|-----------------|----------|----------------|
| Aluminum (Al)   | 4        | 98             |
| Arsenic (As)    | 10       | 84             |
| Antimony (Sb)   | 3        | 95             |
| Barium (Ba)     | 1        | 94             |
| Beryllium (Be)  | 4        | 90             |
| Boron (B)       | 70       | 90             |
| Cadmium (Cd)    | 4        | 96             |
| Calcium (Ca)    | 300      | 95             |
| Cerium (Ce)     | 1        | 84             |
| Chromium (Cr)   | 5        | 93             |
| Cobalt (Co)     | 1        | 88             |
| Gallium (Ga)    | 3        | 88             |
| Germanium (Ge)  | 4        | 84             |
| Lead (Pb)       | 4        | 104            |
| Lithium (Li)    | 3        | 88             |
| Iron (Fe)       | 30       | 102            |
| Magnesium (Mg)  | 3        | 86             |
| Manganese (Mn)  | 3        | 92             |
| Mercury (Hg)    | 30       | 98             |
| Molybdenum (Mo) | 6        | 81             |
| Nickel (Ni)     | 5        | 89             |
| Potassium (K)   | 150      | 113            |
| Phosphorus (P)  | 50       | 82             |
| Silicon (Si)    | .5       |                |
| Sodium (Na)     | 10       | 94             |
| Strontium (Sr)  | 1        | 114            |
| Tin (Sn)        | 10       | 94             |
| Tungsten (W)    | 10       | 89             |
| Vanadium (V)    | 4        | 94             |
| Zinc (Zn)       | 10       | 95             |
| Zirconium (Zr)  | 10       | 85             |

**TABLE 23.** Leachable Trace Metals by ICP-MS.

| Element        | DL ppb (ng/l) | CP5 V-O Only FRPP |
|----------------|---------------|-------------------|
| Aluminum (Al)  | 0.05          | 0.15              |
| Antimony (Sb)  | 0.02          | 1.4               |
| Arsenic (As)   | 0.2           | 0.6               |
| Barium (Ba)    | 0.01          | 2.8               |
| Boron (B)      | 0.05          | *                 |
| Calcium (Ca)   | 0.03          | *                 |
| Chromium (Cr)  | 0.03          | *                 |
| Cobalt (Co)    | 0.02          | *                 |
| Copper (Cu)    | 0.05          | *                 |
| Iron (Fe)      | 0.1           | 0.2               |
| Lead (Pb)      | 0.05          | *                 |
| Lithium (Li)   | 0.03          | *                 |
| Magnesium (Mg) | 0.02          | 0.09              |
| Nickel (Ni)    | 0.05          | *                 |

**TABLE 24. Dynamic Rinse Study of 100 Foot Tubing.**

| Resistivity<br>(megohm-cm) |          | TOC<br>(ppb) |          | Particle Count<br>(particles/ml) |            |            |           |           |
|----------------------------|----------|--------------|----------|----------------------------------|------------|------------|-----------|-----------|
| Minutes                    | Filtrate | Minutes      | Filtrate | Minutes                          | 0.05 $\mu$ | 0.10 $\mu$ | 0.5 $\mu$ | 1.0 $\mu$ |
| 0                          | 11.7     | 6            | 1.02     | 0                                | 58.8       | 18.1       | 6.7       | 3.1       |
| 4                          | 17.0     | 14           | <0.50    | 10                               | 9.6        | 3.6        | 2.7       | 1.0       |
| 6                          | 17.6     | 22           | <0.50    | 20                               | 14.3       | 3.9        | 1.4       | 0.0       |
| 7                          | 17.7     | 30           | <0.50    | 30                               | 6.2        | 0.0        | 1.7       | 0.0       |
| 8                          | 17.8     | 46           | <0.50    | 40                               | 6.9        | 1.9        | 3.1       | 1.7       |
| 9                          | 17.9     | 54           | <0.50    | 50                               | 5.5        | 2.2        | 1.7       | 1.4       |
| 10                         | 18.0     | 69           | <0.50    | 60                               | 3.2        | 0.0        | 0.0       | 0.0       |
|                            |          | 77           | <0.50    | 70                               | 4.1        | 0.0        | 0.0       | 0.0       |
|                            |          | 93           | <0.50    | 80                               | 0.0        | 0.0        | 0.0       | 0.0       |
|                            |          | 100          | <0.50    | 90                               | 1.7        | 0.0        | 0.0       | 0.0       |
|                            |          | 123          | <0.50    | 100                              | 1.9        | 0.0        | 0.0       | 0.0       |
|                            |          | HOURS        |          | 110                              | 3.6        | 1.3        | 1.0       | 0.0       |
|                            |          | 3            | <0.50    | 120                              | 0.0        | 0.0        | 1.0       | 0.0       |
|                            |          | 4            | <0.50    | HOURS                            |            |            |           |           |
|                            |          | 5            | <0.50    | 3                                | 1.6        | 1.3        | 1.0       | 0.0       |
|                            |          | 6            | <0.50    | 4                                | 0.0        | 0.0        | 0.0       | 0.0       |
|                            |          | 7            | <0.50    | 5                                | 0.0        | 0.0        | 0.0       | 0.0       |
|                            |          | 8            | <0.50    | 6                                | 0.0        | 0.0        | 0.0       | 0.0       |
|                            |          | 9            | <0.50    | 7                                | 0.0        | 0.0        | 0.0       | 0.0       |
|                            |          |              |          | 8                                | 0.0        | 0.0        | 0.0       | 0.0       |
|                            |          |              |          | 9                                | 0.0        | 0.0        | 0.0       | 0.0       |

|           | 0 MIN | 10 MIN | 30 MIN | 60 MIN | 2 HRS | END   |
|-----------|-------|--------|--------|--------|-------|-------|
| Fluoride  | <0.05 | <0.05  | <0.05  | <0.05  | <0.05 | <0.05 |
| Chloride  | 0.08  | 0.06   | <0.05  | <0.05  | <0.05 | <0.05 |
| Bromide   | <0.05 | <0.05  | <0.05  | <0.05  | <0.05 | <0.05 |
| Nitrate   | <0.05 | <0.05  | <0.05  | <0.05  | <0.05 | <0.05 |
| Phosphate | <0.05 | <0.05  | <0.05  | <0.05  | <0.05 | <0.05 |
| Sulfate   | <0.05 | <0.05  | <0.05  | <0.05  | <0.05 | <0.05 |
| Lithium   | <0.05 | <0.05  | <0.05  | <0.05  | <0.05 | <0.05 |
| Sodium    | 0.20  | <0.05  | 0.10   | 0.09   | <0.05 | <0.05 |
| Potassium | <0.05 | <0.05  | <0.05  | <0.05  | <0.05 | <0.05 |
| Silica    | <3    | <3     | <3     | <3     | <3    | <3    |
| Calcium   | <0.02 | <0.02  | <0.02  | <0.02  | <0.02 | <0.02 |
| Iron      | <0.02 | <0.02  | <0.02  | <0.02  | <0.02 | <0.02 |

No metals were detected above the DL's.

### CONCLUSION

It is not possible to discuss the myriad of variations of wet chemical procedures that are requested by semiconductor engineers and suppliers. The applications discussed here, however, should illustrate the important role these procedures play for quality control and contamination-free manufacturing. In the future, there will be an even greater need to routinely obtain data from wet chemical procedures as specifications of materials and processes tighten.