

Design, Certification and Verification of Technology for Delivering Sub-ppb, Low-Particle Chemicals to Semiconductor Cleaning Baths in Wafer Fabs

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ABSTRACT

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 (POUs) within the wafer fabrication facility (fab). 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 $\geq 0.2\mu\text{m}$. Several challenges associated with the initial design and installation of the chemical delivery system and their resolution are also described.

INTRODUCTION

It is essential that all reagents in microcircuit production be low in metallic and particulate contamination. It is generally accepted that a large fraction of yield losses in integrated circuit production is due to contamination [1]. In addition, microcircuit geometries are becoming more complex and feature sizes smaller. As a result, processes for fabricating microcircuits are becoming more sensitive to contamination. Impurities which can affect microcircuit device performance may be particulate, organic or metallic. Particulate contamination can result in open or short circuits, crystalline structure defects, altered electrical properties, and unreliable photolithographic reproduction. Metallic contaminants on the semiconductor surface can diffuse into the substrate during subsequent heat treatments, causing drifts in surface potential, current leakage and structural defects in vapor-grown epitaxial layers and reduced breakdown voltage of gate oxides [2].

Chemical delivery systems were developed to meet the chemical management needs of the semiconductor industry. They deliver high purity chemical to points of use within the semiconductor fab without the safety issues associated with the use of bottled chemicals. Well designed systems deliver chemicals with particle concentrations far below those of the incoming chemicals through filtration [3,4]. However, because no technology is currently available to remove metallic contamination, chemical delivery systems must maintain the low impurity level at that of the incoming chemical. As a result, the

metallic impurity levels of the chemical at the POU is no different than the purity of the incoming chemical.

Generally, for certification of acidic and caustic systems, the entire system is first purged with N₂ and flushed with deionized water (DIW). After that, DIW is purged from the system by N₂. Then the system is again filled with DIW and continuously flushed until the particle count is low. The chemical is introduced and allowed to leach in the system until steady state is achieved. For solvent systems, the DIW step is omitted. The system is filled with the solvent after the initial N₂ purging. Samples of the equilibrated chemical are analyzed for metallic impurities after a predetermined number of days. If samples fail to meet the requirements, the chemical is replaced and subsequent samples taken until the system meets requirements.

SYSTEM DESIGN

The chemical delivery systems used at TECH Semiconductor are Model 2500 systems manufactured by FSI International designed to supply sub-ppb chemicals with very low particle concentrations. Design considerations in this equipment for elimination of particulates and metallic extractables and monitoring software capabilities are discussed below.

Very low particle concentrations can be achieved with chemical delivery systems. Microporous membrane filters can effectively remove submicron particulate contamination from process chemicals [6,7]. However, in order to avoid the release of particles associated with sudden changes in flow rate or pressure, flow must be maintained through the filters at all times. Systems with this mode of operation, called Stabilized Distribution™, provide chemicals with extremely low particle concentrations [8].

The FSI Model 2500 system accomplishes Stabilized Distribution by nitrogen pressurization. A simplified system flow schematic of the system is shown in Figure 1. Chemical is transported into the chemical delivery module (CDM) by a high flow double diaphragm pump. Stabilized Distribution is accomplished by continuously flowing chemical from a distribution pressure vessel (DPV) through filters to a supply pressure vessel (SPV1) at atmospheric pressure. Once SPV1 is full of chemical, the flow from the filters is diverted to a second SPV (SPV2) and SPV1 is pressurized with nitrogen to return the chemical to the DPV. When empty, SPV1 is depressurized and prepared to receive chemical when SPV2 is full. Nitrogen pressurization within the system allows continuous, non-pulsing flow through the filter and results in very low particle concentration in the delivered chemical.

The delivery system is based on a compartmental design in which all major components and individual filters are housed in their own compartments. This allows isolation of system components for periodic maintenance or repair without interrupting operation. To minimize the chance of contamination by particles during routine maintenance and monitoring, the drums of incoming chemical, filters and sample ports in the chemical

delivery system are all enclosed in Class 100 HEPA filtered cabinets. The filters installed in all delivery systems were 0.05 μ m PF80 filters. They consist of all-Teflon membrane filters in a stacked disc configuration enclosed in a PFA housing. Typically 5 to 9 filters are used in parallel, depending on the viscosity of the chemical.

Many chemicals supplied by delivery systems can extract metallic contaminants from system components and degrade the quality of the chemical. ChemFill delivery systems are designed such that only perfluoroalkoxy (PFA) or polytetrafluoroethylene (PTFE) parts contact the chemical because these materials have low metallic extractables [9]. The SPVs are injection molded PFA with a polyvinylchloride outer shell for increased pressure capability. The DPV is constructed from 304 stainless steel lined with PFA. The chemical transfer pump is a double diaphragm pump with no exposed metal parts. Metal components in the pump are separated from the chemical by the diaphragm. If the diaphragm fails, a leak detector immediately isolates the pump from the chemical. A modified remote shuttle assembly greatly improves pump reliability. Since the majority of metallic ions extracted from delivery systems originate in component manufacturing processes and not in the resins, proper selection of delivery system components is critical to maintaining high purity chemical [10].

Each of the chemicals delivered has individual secondary containment. Tubing is routed so that all mechanical connections to filters, valves, pressure vessels and sampling ports are located inside containment boxes. Access to the secondary containment is interlocked, causing system shutdown when containment boxes are opened. Distribution tubing is made of PFA with clear PVC secondary containment to allow visual inspection and is sloped toward a containment box with a chemical trap and leak sensor. As an added safety precaution, the delivery system is constructed so that any leaked chemical will eventually drain back to the CDM.

Each central delivery module is a "stand alone" unit, complete with its own power supply and control system. The 486-based control system provides complete information regarding set-up, control, operation and maintenance functions, even during abnormal shut down. All aspects of the chemical delivery module's operation, including POU dispense functions, can be controlled using the interactive user interface panel. Through the interface panel, the controller provides data and trending for critical component cycles, filter usage, system pressures, and chemical demand, as well as status of sampling, self diagnostic and maintenance routines.

The chemical management system at TECH Semiconductor supplies 15 chemicals to the wafer fab. Two of the chemicals, 0.49% and 2.5% HF are prepared on site from 49% HF and DI water by using FSI blending systems. The blending operation and performance of these systems are described in detail elsewhere [11].

CONTROL OF INCOMING CHEMICAL PURITY

For incoming chemical quality assurance, chemicals from commercially available vendors were examined at the local warehouse. Two drums were sampled and analyzed for each batch of chemical. The batch was only accepted when both drums met requirements based on metallic impurities. Dip tubes were inserted into the drums in a class 10 clean hood before delivery of chemicals to TECH Semiconductor. Subsequently, the chemicals were stored and consumed on a first in first out basis. Table I shows the incoming quality control (IQC) data of chemicals used at TECH Semiconductor. For important process cleaning chemicals such as HF, NH₄OH, H₂O₂ and IPA, only impurity levels of less than 1 ppb per contaminant were accepted.

Table I: Incoming quality control data of chemicals used at TECH Semiconductor

Chemical	Ca	Fe	K	Na	Zn
Acetone	0.1-0.4	0.1-0.5	0.1-7.1	0.1-1.4	0.1-13.3
BHF	0.1-0.4	0.1-0.9	0.1-0.3	0.1-0.6	0.1-0.5
HCl	0.4-3.8	0.1-1.8	0.1-0.8	0.1-0.5	0.1-0.9
HF	0.1-0.7	0.1-0.6	0.1-0.9	0.1-0.9	0.1-0.6
H ₃ PO ₄	10.6-37.5	107-211	7.2-48.3	44.7-139	-----
HNO ₃	0.2-3.5	0.3-3.1	0.1-1.0	0.1-2.2	0.1-3.0
H ₂ O ₂	0.1-1.5	0.1-1.0	0.1-0.6	0.1-1.0	0.1-1.0
H ₂ SO ₄	0.1-2.9	0.1-0.5	0.1-0.5	0.1-4.6	0.1-2.2
IPA	0.1-0.6	0.1-0.4	0.1-1.1	0.1-4.5	0.1-2.9
NH ₄ OH	0.1-3.4	0.1-0.3	0.1-0.6	0.1-0.5	0.1-1.6

SYSTEM CERTIFICATION AND MONITORING PROCEDURES

A newly installed chemical delivery system must meet all purity requirements to ensure that the system can supply high quality chemicals before it is certified for use. This process involves system start up, implementation of system monitoring, sampling and analysis procedures, and identification and resolution of any problems that might arise. After certification, a continuous monitoring program of system performance assures both system operation reliability and a sustained high quality chemical supply.

For certification, the system was filled with chemical for a short period of time to dissolve the bulk of any contamination present. The system was drained completely and refilled with chemical. This chemical remained in the system for an extended time to leach additional impurities. The system was then drained and refilled a third time. Certification samples were taken after this chemical was in the system for 24 hours or more. The certification plan used at TECH Semiconductor involved ensuring chemical quality at the points shown in Table II.

Table II: Summary of the delivery system sampling locations

System	49% HF	NH ₄ OH	H ₂ O ₂	HCl	HNO ₃	H ₂ SO ₄	H ₃ PO ₄	IPA	Acetone
CDM	1	1	1	1	1	1	1	1	1
Submain	5	6	6	4	5	3	1	6	2

For continuous monitoring, samples were drawn from both the CDM filters (location S in Figure 1) and at equipment POU's. The CDM sampling schedule shown in Table III is used for continuous routine monitoring after certification.

Table III: Schedule for routine monitoring of delivery systems after certification

System	Sampling Frequency
0.49% HF	1 week
2.5% HF	1 week
49% HF	1 week
H ₂ O ₂	1 week
NH ₄ OH	1 week
H ₂ SO ₄	2 weeks
HNO ₃	2 weeks
HCl	2 weeks
IPA	2 weeks
Acetone	3 weeks
H ₃ PO ₄	6 weeks

The sample bottles used were thoroughly cleaned with HNO₃ and DI water prior to taking samples. The bottles were equilibrated with the sample to be taken by rinsing them three times with the chemical just prior to sampling. The samples were collected from the filter compartments into the bottles under Class 100 clean environment conditions and submitted for analysis.

The metallic ion concentrations were analyzed using a VG PlasmaQuad STE II Plus Inductively Coupled Plasma-Mass Spectrometer and a Varian 300/400 Graphite Furnace Atomic Absorption Spectrophotometer. The accuracy of both these instruments is ± 0.1 ppb for each element.

Particulate contamination was monitored at the outlets of the CDM filters using Hiac Royco μ Count02 particle counters which have a minimum detectable particle diameter of $0.2\mu\text{m}$. When the particle counts were taken, the only flow through the filter was that going through the particle counter. Since the flow through the filters is usually much higher than the 100 ml/min supplying the counter and since filter performance improves with increasing flow, system particle concentrations are lower than those indicated by this test.

DELIVERED CHEMICAL PURITY

System Certification

Four problems were encountered during system certification. Two of the problems resulted in high particle concentrations, two in high metal ion concentrations. A discussion of these problems and their resolution is presented here.

Upon start up, the particle counts in buffered oxide etch (BOE) were approximately 2000/ml for particles $\geq 0.20\mu\text{m}$ in diameter, against the system specification of 3 particles/ml. The high particle counts were accompanied by a high pressure drop across the filters. The combination of problems indicated that the hydrophobic filters had spontaneously dewet in BOE and that the chemical stream contained bubbles [12]. Replacement of the Teflon $0.05\mu\text{m}$ filters with $0.2\mu\text{m}$ hydrophilic filters reduced the system pressure drop and particle counts, as shown in Table IV. Subsequent installation of $0.1\mu\text{m}$ hydrophilic filters further reduced the particle counts and resulted in <1 particle/ml $\geq 0.20\mu\text{m}$ at the BOE system outlet.

Table IV: The impact of filter type on BOE system particle output

Filter Type	Pore Size	Particles $\geq 0.20\mu\text{m}$
Hydrophobic	$0.2\mu\text{m}$	$\sim 2000/\text{ml}$
Hydrophilic	$0.2\mu\text{m}$	$\sim 5/\text{ml}$
Hydrophilic	$0.1\mu\text{m}$	$<1/\text{ml}$

When the phosphoric acid system was started up, the particle concentrations at the POU were in excess of 100 particles/ml ($\geq 0.20\mu\text{m}$), against the system specification of 30/ml. For troubleshooting, particle concentrations were measured at the outlet of each of the nine filters in the system. All of the filters except one showed similar low particle concentrations $<30/\text{ml}$. The last filter produced chemical containing approximately 200 particles/ml. When the faulty filter was replaced, the chemical at the POU contained 6 particles/ml ($\geq 0.20\mu\text{m}$), well below the design specification.

Metallic ion analyses of the HCl system revealed that the iron concentration was erratic and above specification when the system was first operated. Examination of the system

revealed that HCl leakage from a single faulty component had caused corrosion of an external metal part. Iron from the corrosion had diffused into the chemical flow stream, contaminating the HCl. Figure 2 shows the level of iron contamination in the HCl and the reduction when the faulty component was replaced. When the system was subsequently flushed free of contamination, it remained consistently within iron specification.

The fourth problem encountered was caused by an improper seal in the blending system. The seal allowed HF to attack stainless steel in a sensor. Iron, extracted from the stainless steel, diffused into the chemical in the system. Figure 3 shows that the iron concentration was high and erratic, while all other metal ion concentrations were consistently low. Installation of a sensor with a redesigned seal eliminated the leakage problem and accompanying iron contamination [11].

Once these four problems were resolved, the chemical quality was reconfirmed at the sampling locations shown in Table II. In Table V, actual data for 10 key metals are provided for 9 chemicals. Particle data at 3 different particle sizes are included as well. When total metals are considered, the total detection limit (TDL) is 3.4 ppb (34 elements at 0.1 ppb DL each) for all chemicals except IPA and acetone, which have TDLs of 2.4 ppb and 2.2 ppb, respectively. These are the lowest concentrations attainable as it was assumed that elements not detected were present at their detection limit. Table V indicates that the total metal concentrations in the systems varied from 3-12 ppb. These concentrations are only 1 to 9 ppb above the detection limits of 2 to 3 ppb. Notable exceptions were calcium in ammonium hydroxide and zinc in acetone. These deviations were a result of contamination in the incoming chemical, hence discounted.

System Monitoring

After certification, a continuous monitoring program was implemented based on the sampling schedule shown in Table III. In Table VI, typical data for 10 key metallic contaminants in 9 systems are provided over more than 6 months time. A comparison of Tables V and Table VI indicates that the concentration of most metallic contaminants was maintained or decreased with time. The trend toward lower concentrations is believed to be the combined result of cleaner incoming chemical, improved sampling and analysis techniques and reduced extraction from the system.

In Figure 4, the total metals of four representative chemicals (one each: acid, base, oxidizer and solvent) are illustrated. The mean total metals concentrations in the three chemicals were 4-7 ppb, which is only 1-4 ppb above the TDL. The 3 sigma upper control limits were 6-12 ppb, 3-9 ppb above the TDL. These numbers are typical of the performance of the other delivery systems, as shown in Table VI.

The monitoring program has ensured that the delivery systems continue to provide high quality chemical following certification. Figure 5 shows an example of a system which has operated very smoothly over the monitoring period to date. Iron concentrations in this system have remained consistently below 1 ppb. Most elements in most of the

system have behaved in this manner. However, several problems have arisen which have been identified and resolved by this procedure. Figure 2 indicates that the iron concentrations in HCl increased to 3-5 ppb around day 320. The increase occurred because the quick connect between the drum and the CDM had been inadvertently contaminated. Cleaning the connection resolved the problem quickly. Not all of the issues were this easily resolved.

The particle monitoring data shown in Table VI indicate the average particle performance at 3 different particle sizes since certification. Particle concentrations were typically $<1.5/\text{ml} \geq 0.2\mu\text{m}$ with the exception of difficult to filter chemicals like sulfuric and phosphoric acids, which typically contained 5-15 particles/ml. In Figure 6, particle concentrations for 4 representative chemicals are shown. No apparent trends are evident. Monitoring will help determine when the filters fail.

SUMMARY

On-site chemical delivery systems were designed and built by FSI and installed at TECH Semiconductor. These systems have demonstrated very pure chemical delivery achieved through careful system design and materials selection. Certification procedures and 6 months of continuous system monitoring have demonstrated that most of the chemicals supplied contain <1 ppb of most metallic elements and <3 particles/ml ($\geq 0.20 \mu\text{m}$) at the fab points of use.

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Table V: Typical metallic and particle impurity data for chemicals delivered by the chemical delivery systems during system certification

	70% HNO ₃	37% HCl	49% HF	30% H ₂ O ₂	29% NH ₄ OH
Al, ppb	0.56	0.43	0.82	0.59	0.31
Ca, ppb*	1.48	1.54	0.50	1.01	1.36
Cr, ppb	0.11	0.10	0.11	0.10	0.10
Cu, ppb	0.34	0.10	0.10	0.21	0.10
Fe, ppb*	1.41	4.08	0.78	0.64	0.28
K, ppb*	0.51	0.16	0.20	0.32	0.31
Mg, ppb	0.23	0.16	0.27	0.12	0.10
Na, ppb*	1.06	0.54	0.47	1.63	0.58
Ni, ppb	0.17	0.13	0.10	0.29	0.13
Zn, ppb	0.46	0.26	0.17	0.45	0.43
Total Metallics, ppb	9.40	9.76	6.47	7.79	6.44
Particles/ml					
≥0.20μm	2.93	1.38	1.41	1.33	1.44
≥0.30μm	1.11	0.31	0.54	0.47	0.41
≥0.50μm	0.29	0.14	0.17	0.15	0.09

	96% H ₂ SO ₄	Isopropyl Alcohol	Acetone	85% H ₃ PO ₄
Al, ppb	0.50	0.11	0.20	16
Ca, ppb*	0.75	0.29	0.30	86
Cr, ppb	0.13	0.10	0.10	-
Cu, ppb	1.10	0.15	0.13	10
Fe, ppb*	0.84	0.57	0.17	140
K, ppb*	0.21	0.36	0.73	5
Mg, ppb	0.25	0.14	0.10	<10
Na, ppb*	0.73	1.54	1.33	35
Ni, ppb	0.10	0.10	0.10	-
Zn, ppb	0.22	0.72	6.70	-
Total Metallics, ppb	7.78	5.78	11.50	-
Particles/ml				
≥0.20μm	6.07	1.37	1.28	6.01
≥0.30μm	2.11	0.70	0.29	0.92
≥0.50μm	0.65	0.10	0.05	0.04

* Measured by graphite furnace atomic absorption; all others by inductively coupled mass spectroscopy.

Table VI: Typical metallic and particle impurity data for chemicals delivered by the chemical delivery systems during routine monitoring

	70% HNO ₃	37% HCl	49% HF	30% H ₂ O ₂	29% NH ₄ OH
Al, ppb	0.17	0.37	0.28	0.57	0.30
Ca, ppb*	0.90	1.31	0.21	0.35	0.44
Cr, ppb	<0.10	0.15	<0.10	0.10	0.11
Cu, ppb	0.19	<0.10	0.11	0.11	0.13
Fe, ppb*	1.25	1.43	0.22	0.29	0.13
K, ppb*	0.46	0.15	0.14	0.27	0.14
Mg, ppb	0.11	0.27	0.11	0.17	0.14
Na, ppb*	0.36	0.27	0.17	0.86	0.29
Ni, ppb	0.13	0.12	0.14	0.12	0.18
Zn, ppb	0.94	0.40	0.18	0.22	0.19
Total Metallics, ppb	8.06	7.04	6.20	5.80	4.66
Particles/ml					
≥0.20μm	-	0.42	0.08	0.77	1.50
≥0.30μm	0.15	0.08	0.01	0.22	0.17
≥0.50μm	0.01	0.02	<0.01	0.04	0.02

	96% H ₂ SO ₄	Isopropyl Alcohol	Acetone	85% H ₃ PO ₄
Al, ppb	0.36	0.13	0.13	-
Ca, ppb*	0.69	0.21	0.16	48
Cr, ppb	<0.10	<0.10	<0.10	-
Cu, ppb	0.12	0.10	0.13	-
Fe, ppb*	0.22	0.12	0.12	128
K, ppb*	0.11	0.24	1.82	10
Mg, ppb	<0.10	0.20	0.11	-
Na, ppb*	0.23	2.08	0.47	70
Ni, ppb	<0.10	0.10	0.12	-
Zn, ppb	0.23	0.87	0.86	-
Total Metallics, ppb	4.98	5.57	5.34	-
Particles/ml				
≥0.20μm	5.05	0.69	0.38	14.9
≥0.30μm	1.90	0.14	0.07	3.8
≥0.50μm	0.06	0.01	0.02	0.02

* Measured by graphite furnace atomic absorption; all others by inductively coupled mass spectroscopy.

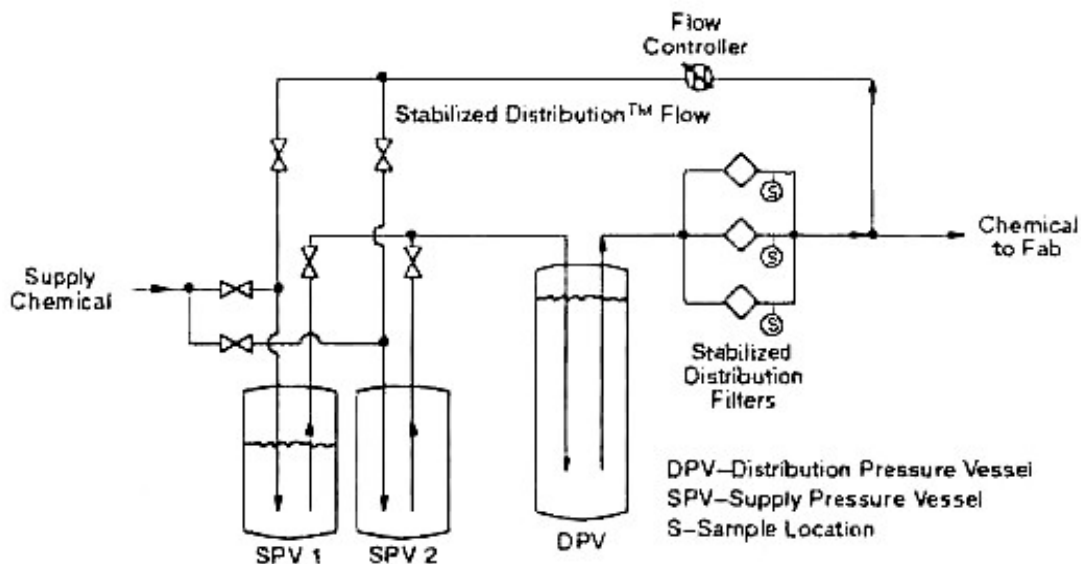


Figure 1: ChemFill Model[®] 2500 Simplified Flow Schematic

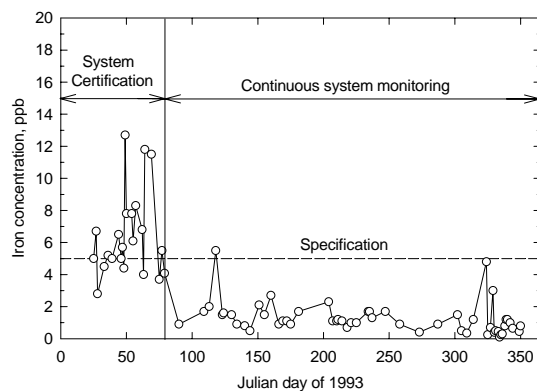


Figure 2: Iron concentrations in 37% HCl at the CDM filter outlet

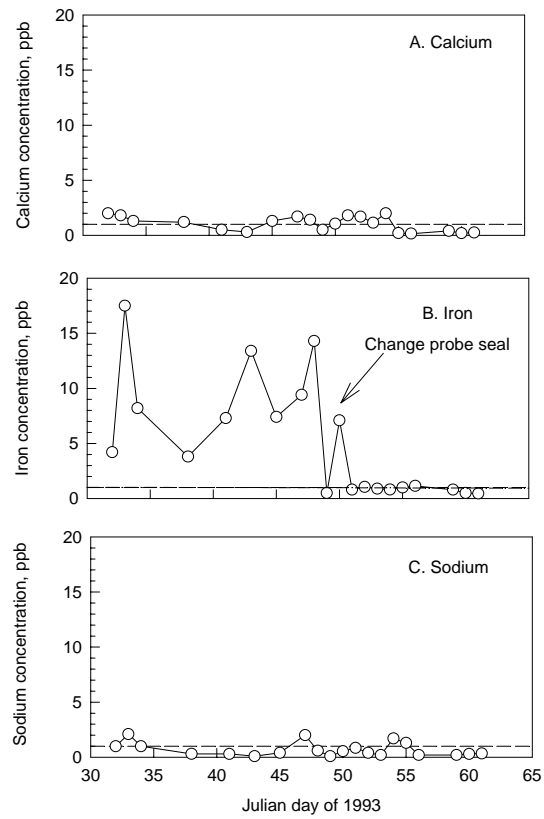


Figure 3: Metal ion concentrations in 0.49% HF before certification

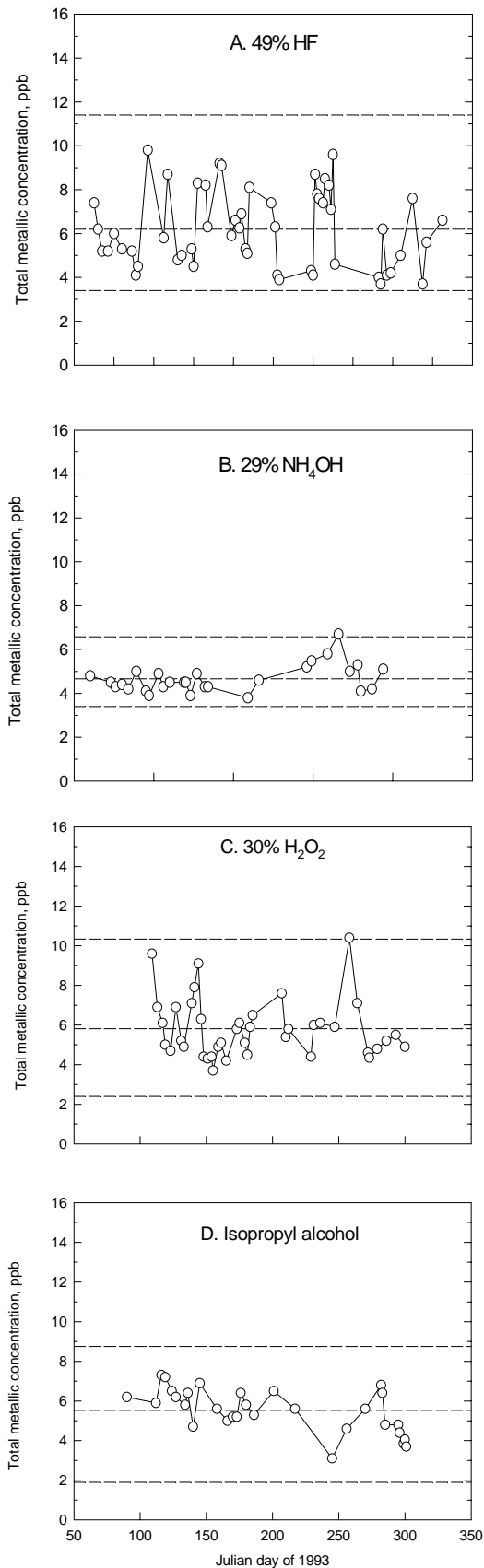


Figure 4: Total metal concentrations (34 elements) in representative chemicals

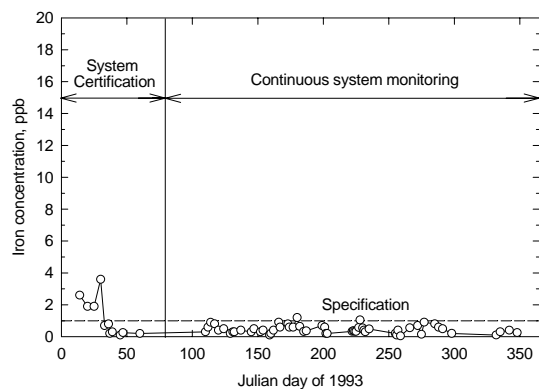


Figure 5: Iron concentrations in 49% HF at the CDM filter outlet

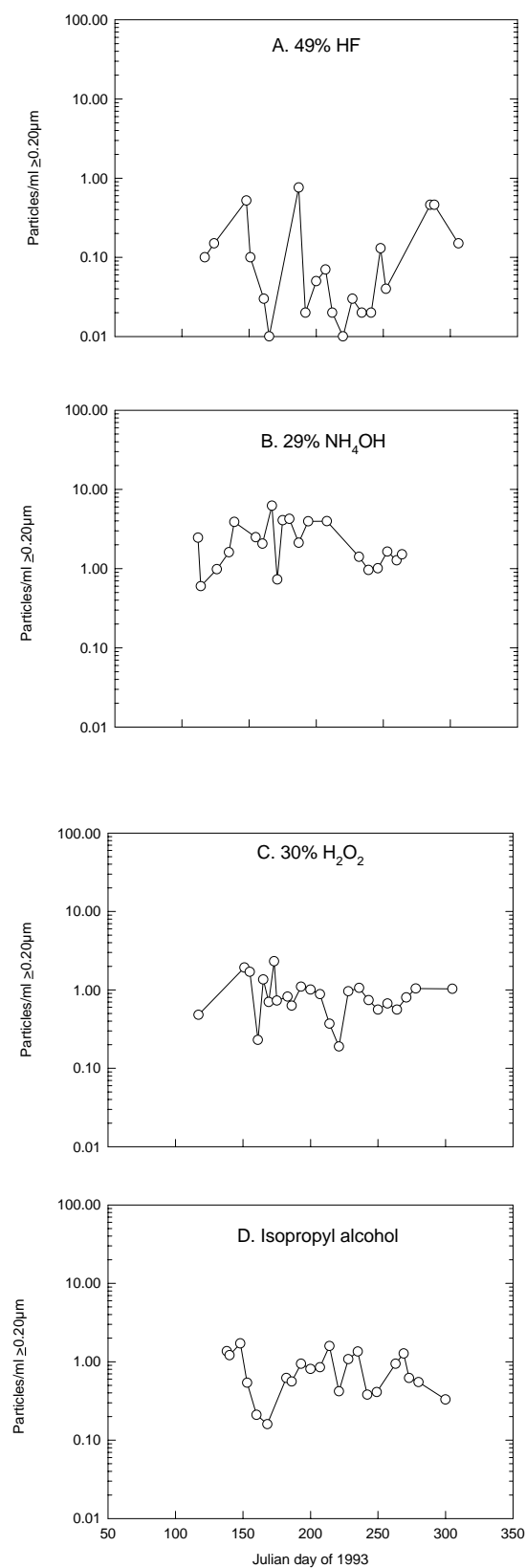


Figure 6: Particle concentrations in representative chemicals