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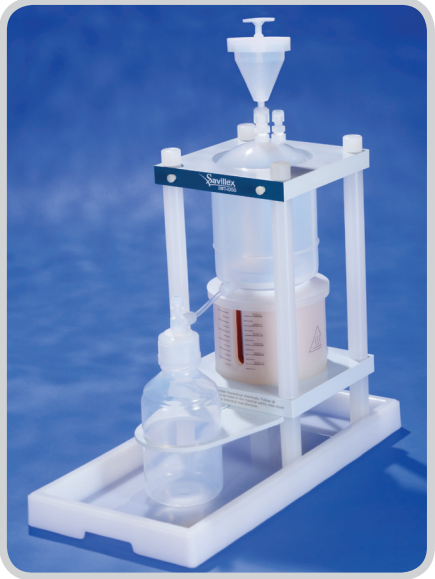
Savillex Technical Note

Performance of the DST-1000 Acid Purification System

Summary

The Savillex DST-1000 Acid Purification System is a sub-boiling still manufactured from PFA that produces high purity acid from cheaper, lower grade acids. A new DST-1000 was installed inside a fume hood in a semiconductor clean room and used for the purification of trace metal grade (1 ppb) HF. The first 15 batches of HF produced by the DST-1000 from trace metal grade were analyzed for trace metals using ICP-MS. For comparison, high purity grade (10 ppt) HF was purchased and also analyzed. By the 15th batch, the HF produced by the DST-1000 was equivalent in quality to commercial high purity grade. Trace metal data for purified HNO3 and HCL is also given. All acid was produced using the DST-1000 controller on HIGH setting, which produces 500 mL of high purity acid in approx. 15 hours. The DST-1000 has been shown to produce very high quality acid, at a fraction of the cost of commercially available high purity grade. In addition, since the DST-1000 produces fresh acid on demand, the degradation in quality over time with commercial high purity acid (as the bottle is repeatedly opened) is avoided.



Savillex DST-1000 Acid Purification System

Methodology

The work described in this note was performed at a semiconductor R&D lab. All cleaning, sample collection, and sample analysis was performed in a Class 1 clean room, maintained at a constant temperature of 20.4˚ C. The DST-1000 was placed in a fume hood, while the controller was outside the hood. The DST-1000 was new and unused, so that the initial quality of the acid produced, and time taken to reach high purity, could be measured. Initial cleaning following the recommend procedure was performed: following unpacking, but prior to connecting the outlet tubing to the collection bottle, 500 mL of HF was placed in the DST-1000 and the unit was operated with the temperature controller set to HIGH for 48 hours. The unit is initially cleaned by the refluxing acid, which is then discarded. This procedure was repeated with DI water and the unit was then ready for use. The outlet and collection bottle were connected, and 15 successive distillations (batches) of 1 L trace metal grade HF were performed and collected. In all cases the controller was set to HIGH (fastest distillation rate). At the end of each batch, the remaining acid (approx. 50 mL) in the DST-1000 was drained and discarded. Trace metal grade HNO3 and HCl acid were purified on separate DST-1000s (also on HIGH setting), which had been previously used. *Note: if multiple acids are to be purified, it is recommended to dedicate a separate DST-1000 for each acid.*

Analysis - HF

Trace metal analysis was performed on the 1st, 5th and 15th batches of HF produced. Replicate samples of each batch were preconcentrated using an in-house preconcentration system, along with replicates of a new bottle of commercial high purity HF and trace metal grade HF. A blank and spike recovery were also prepared with each replicate. The samples were then analyzed using an Agilent 7500cs ICP-MS.

Table 1 shows the detection limits reported using the preconcentration technique, and analytical data for the commercial high purity (10 ppt) grade HF, trace metal (1 ppb) grade HF and the 3 distilled batches.

Analyte	Detection Limit (ppt)	High Purity Grade HF (10 ppt)	Trace Metal Grade HF (1 ppb)	Distilled HF (1st batch)	Distilled HF (5th batch)	Distilled HF (15th batch)
Li	*1*	*<1*	*<1*	*<1*	*<1*	*<1*
Na	*1*	*<1*	*205*	*49*	*14*	*6*
Mg	*1*	*2*	*27*	*5*	*1*	*<1*
Al	*1*	*8*	*113*	*205*	*15*	*6*
K	*1*	*3*	*35*	*45*	*5*	*3*
Ca	*1*	*9*	*39*	*78*	*7*	*2*
Cr	*1*	*1*	*41*	*10*	*1*	*3*
Fe	*1*	*1*	*120*	*312*	*7*	*9*
Ni	*1*	*<1*	*65*	*7*	*<1*	*1*
Cu	*1*	*<1*	*43*	*9*	*<1*	*2*
Zn	*1*	*2*	*19*	*5*	*1*	*3*
W	*1*	*<1*	*21*	*7*	*5*	*2*
Mo	*1*	*3*	*2*	*6*	*4*	*1*
Ti	*1*	*<1*	*67*	*16*	*6*	*6*
Co	*1*	*<1*	*21*	*1*	*<1*	*<1*
Ge	*1*	*<1*	*<1*	*<1*	*<1*	*<1*
Sb	*1*	*<1*	*<1*	*<1*	*<1*	*<1*
Rh	*1*	*<1*	*<1*	*<1*	*<1*	*<1*
Be	*1*	*<1*	*<1*	*<1*	*<1*	*<1*
V	*1*	*<1*	*<1*	*1*	*<1*	*<1*
Mn	*1*	*<1*	*6*	*1*	*<1*	*<1*
Zr	*1*	*<1*	*2*	*3*	*<1*	*<1*
Ag	*1*	*<1*	*<1*	*<1*	*<1*	*<1*
Cd	*1*	*<1*	*<1*	*<1*	*<1*	*<1*
Sn	*1*	*<1*	*2*	*1*	*1*	*<1*
Cs	*1*	*<1*	*<1*	*<1*	*<1*	*<1*
Ba	*1*	*<1*	*4*	*<1*	*<1*	*<1*
Hf	*1*	*<1*	*<1*	*<1*	*<1*	*<1*
Ta	*1*	*<1*	*<1*	*<1*	*<1*	*<1*
Pb	*1*	*<1*	*1*	*<1*	*<1*	*<1*

Table 1. *Analytical data for HF obtained using ICP-MS. All data in ppt.*

As can be seen, by the 5th batch, the distilled HF produced by the DST-1000 is very close to the quality of the commercial high purity HF, and by the 15th batch is essentially of equivalent purity. The analysis of trace metal grade HF shows the amount of trace contaminants removed by the DST-1000.

Analysis - HNO_3 and HCl

The analysis of DST-1000 HNO3 and HCl was performed separately using different DST-1000 units. As before, replicate samples of each acid were preconcentrated using an in-house preconcentration system, along with replicate samples from new bottles of commercial high purity (10 ppt grade) HNO_3 and HCl. A blank and spike recovery were also prepared with each replicate to check the quality of the measurements. Analysis was performed using both Agilent 7500cs ICP-QMS and Agilent 8800 ICP-QQQ. The detection limits reported using the preconcentration technique, along with quantitative data for the commercial high purity acid and DST-1000 produced acid are shown in tables 2 and 3. For the large majority of elements, the DST-1000 produced acid is actually cleaner than the commercial high purity grade – even though the commercial acid bottle was opened for the first time. For HCl, Hg was also measured to assess the suitability of DST-1000 produced acid for cold vapor AA. As can be seen, the DST-1000 is able to remove Hg from HCl, making it well suited for cold vapor AA use.

Analyte	Detection Limit	High Purity Grade HNO_3 (10 ppt grade)	DST Produced HNO_3
Li	*1*	*<1*	*<1*
Na	*1*	*1*	*<1*
Mg	*1*	*2*	*<1*
Al	*1*	*1*	*<1*
K	*1*	*<1*	*<1*
Ca	*1*	*<1*	*<1*
Cr	*1*	*5*	*1*
Fe	*1*	*7*	*4*
Ni	*1*	*7*	*1*
Cu	*1*	*6*	*4*
Zn	*1*	*<1*	*<1*
W	*1*	*<1*	*<1*
Hf	*1*	*<1*	*<1*
Mn	*1*	*<1*	*<1*
Ti	*1*	*<1*	*<1*
Co	*1*	*<1*	*<1*
Ge	*1*	*<1*	*<1*
Sb	*1*	*<1*	*<1*
Te	*1*	*<1*	*<1*
Ag	*1*	*<1*	*<1*
Au	*1*	*<1*	*<1*
Gd	*1*	*<1*	*<1*
La	*1*	*<1*	*<1*
Pt	*1*	*<1*	*<1*
Sr	*1*	*<1*	*<1*
Zr	*1*	*<1*	*<1*
In	*1*	*<1*	*<1*
Mo	*1*	*2*	*<1*
Ta	*1*	*<1*	*<1*
Be	*1*	*<1*	*<1*
V	*1*	*1*	*1*
As	*1*	*2*	*2*
Cd	*1*	*<1*	*<1*
Cs	*1*	*<1*	*<1*
Ba	*1*	*<1*	*<1*
Pb	*1*	*<1*	*<1*

Table 2. *Analytical data for HNO_3 obtained using ICP-MS. All data in ppt.*

Analyte	Detection Limit	High Purity Grade HCl (10 ppt grade)	DST Produced HCl
Li	1	<1	<1
Na	1	<1	7
Mg	1	<1	<1
Al	1	1	6
K	1	<1	2
Ca	1	2	4
Cr	1	5	3
Fe	1	20	17
Ni	1	29	2
Cu	1	1	4
Zn	1	7	1
W	1	<1	<1
Hf	1	<1	<1
Mn	1	<1	<1
Ti	1	130	1
Co	1	11	<1
Te	1	<1	<1
Ag	1	<1	<1
Au	1	<1	<1
Gd	1	<1	<1
La	1	<1	<1
Pt	1	<1	<1
Sr	1	<1	<1
Zr	1	<1	<1
In	1	<1	<1
Mo	1	1	2
Ta	1	<1	<1
Be	1	<1	<1
V	1	<1	<1
As	1	37	6
Cd	1	<1	<1
Cs	1	<1	<1
Ba	1	<1	<1
Pb	1	<1	<1
Hg	1	1	<1

Table 3. *Analytical data for HCl obtained using ICP-MS. All data in ppt.*

Distillation Rates

The DST-1000 controller has 3 temperature settings: LOW, MEDIUM and HIGH. These control the temperature (and therefore distillation rate) by adjusting the amount of time at which power is supplied to the heater. At HIGH setting, the heater is on continuously, and the temperature of the acid is approx. 90 deg˚C (sub-boiling conditions are maintained). This means that the DST-1000 cannot be set to too high a temperature, which would cause boiling and allow contaminants to be transferred with the distilled acid. Following the initial HF distillations and sample analysis, production rates for HF, HNO_3 and HCl were calculated over a 6 hour time period using the different controller settings. These are shown in table 4: different mineral acids distil at slightly different rates. On the HIGH setting, 500 mL of acid is distilled in 12-15 hours depending on the acid. Operated overnight, the DST-1000 will produce 500 mL of high purity acid ready for use each morning.

Acid	Controller Setting	Total Distillate (mL)	Distillation Rate (mL/hr)
HF	*HIGH*	*200*	*33.3*
HF	*MEDIUM*	*75*	*12.5*
HF	*LOW*	*50*	*8.3*
HNO_3	*HIGH*	*250*	*41.7*
HNO_3	*MEDIUM*	*120*	*20*
HNO_3	*LOW*	*46*	*7.7*
HCl	*HIGH*	*225*	*37.5*
HCl	*MEDIUM*	*109*	*18.2*
HCl	*LOW*	*38*	*6.3*

Table 4. *Distillation rates for various acids at different controller settings.*

From the data shown here it is unlikely that acid quality can be further improved by using the low distillation rates, (LOW and MEDIUM settings), however these settings are useful for operating the DST-1000 over a weekend without the system evaporating to dryness. A great benefit of the DST-1000 is that fresh high purity acid is always available. Commercial high purity acid, while of excellent quality when first opened, deteriorates each time the bottle is opened.

User Observations

The following comments on the DST-100 were made by the semiconductor chemists who performed the work described in this note.

- Initial set-up and cleaning, following the Savillex manual was simple and efficient
- No additional facilities were required (i.e. water cooling) to operate the unit. This simplifies and improves the overall value of the unit.
- The overall footprint is small and welcomed since hood space is precious in a cleanroom lab
- HNO_3 does not discolor during the distillation process - we have observed this with other distillation systems
- The reservoir is simple and safe to fill
- The slower distillation rate settings are convenient for weekend or 24 hour operation
- Low maintenance after initial cleaning. Recommend dedicating a DST-1000 to only one acid type.

Note: This lab now has four DST-1000 systems, operating continually.

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