

Sample tests for stain problem at KEK

LCWA2009 at Albuquerque

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Introduction: Stain problem at STF/KEK

We replaced the EP acid of EP-tank at STF/KEK recently. After the replacement, cavity performances (MHI#6, MHI#7, MHI#8) become very bad (Eacc<20 MV/m) limited by heavy field emissions.

We observed brown spots and traces inside the 9-cell cavities (MHI#6, MHI#7, MHI#8);

Examples: (red indicates bad)

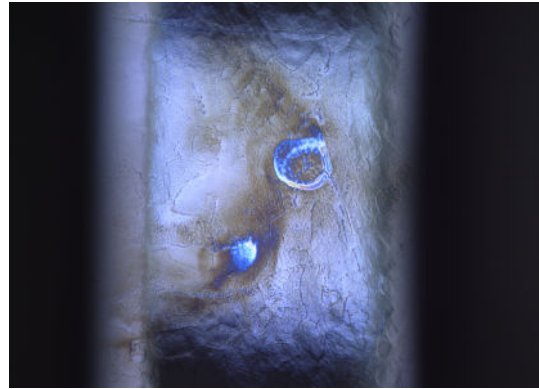
MHI#06 : spot or traces on BP-#1, cell#1, cell#2, cell#3, cell#4, cell#8, cell#9, #9-BP

MHI#07 : BP-#1, cell#1, cell#2, cell#3, cell#4, cell#5, cell#6, cell#7, cell#8, cell#9, cell#9-BP

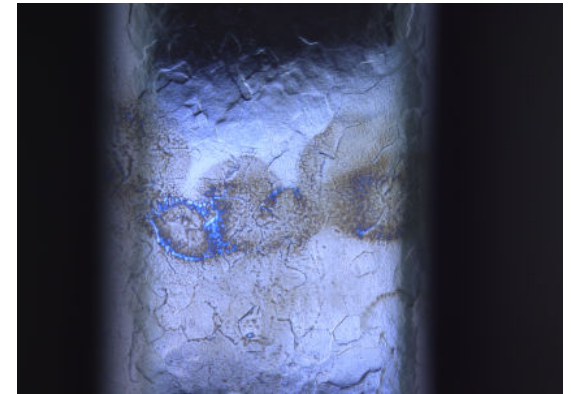
MHI#6 1-cell equator, t = 306 deg.
Downstream : Outside weld area



MHI#6 #9-BP, t = 241 deg. -1



MHI#6 #9-BP, t = 241 deg. -2



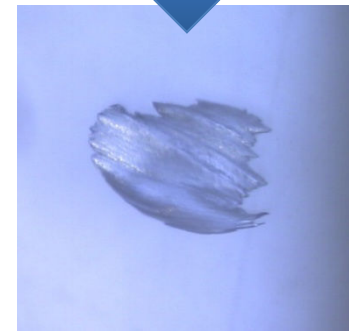
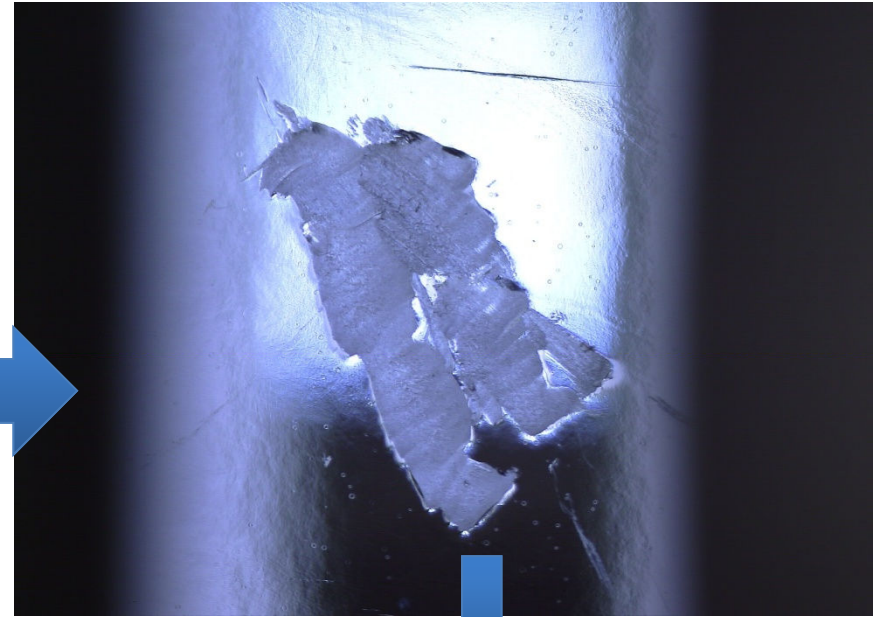
Kyoto-camera pictures using new LED illumination

For more details about the vertical test results of these cavities, see the presentation "KEK cavity preparation for S1G" on 30th Sept. by E. Kako (KEK).

Scraping the brown spot by scraper

Scraped the brown spot by scraper.

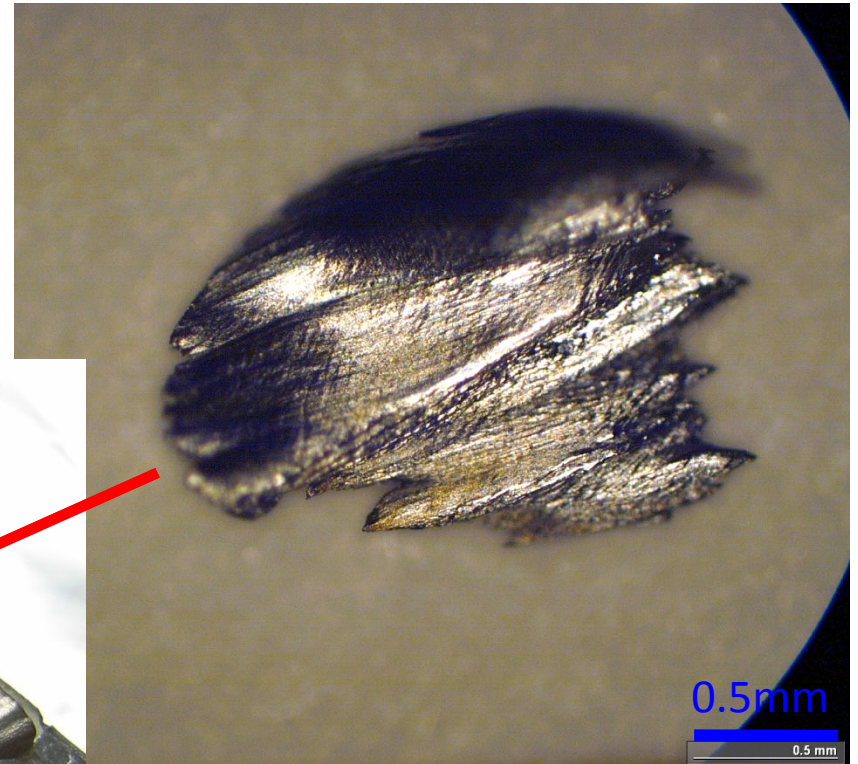
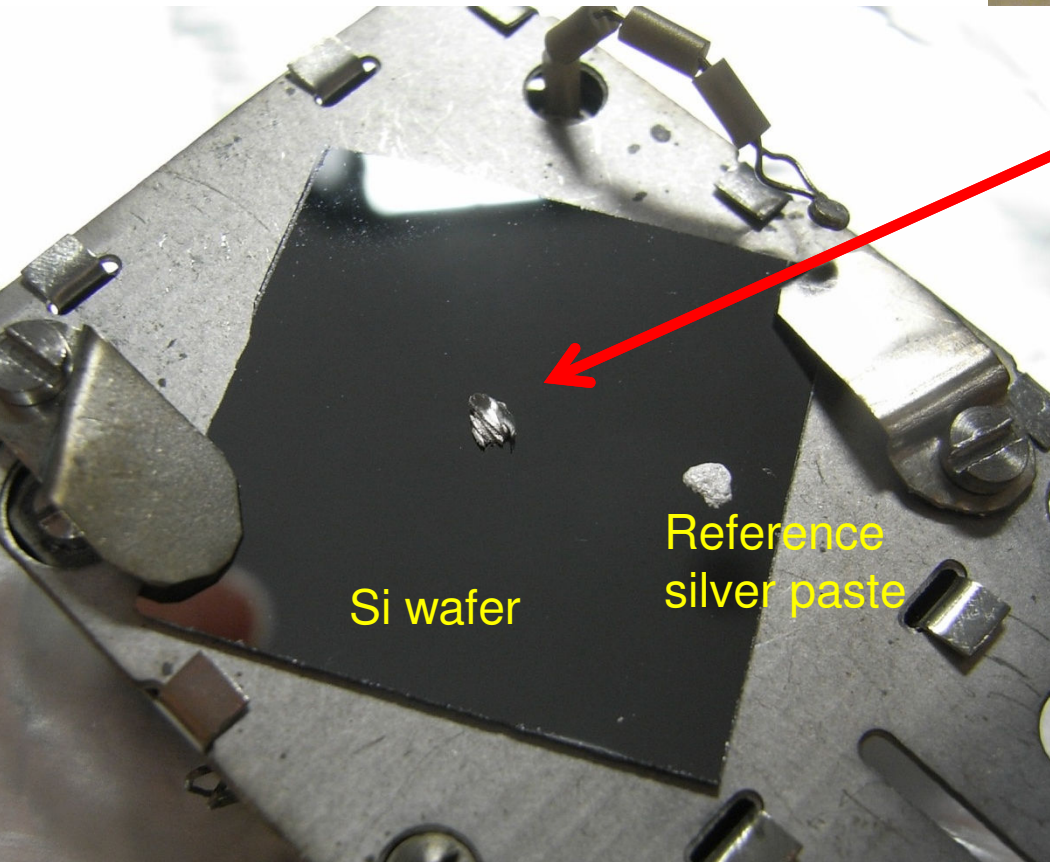
An old MHI#0 9-cell cavity was EP'ed.



A brown spot was found inside the beam-pipe of old MHI#0 9-cell cavity after the EP.

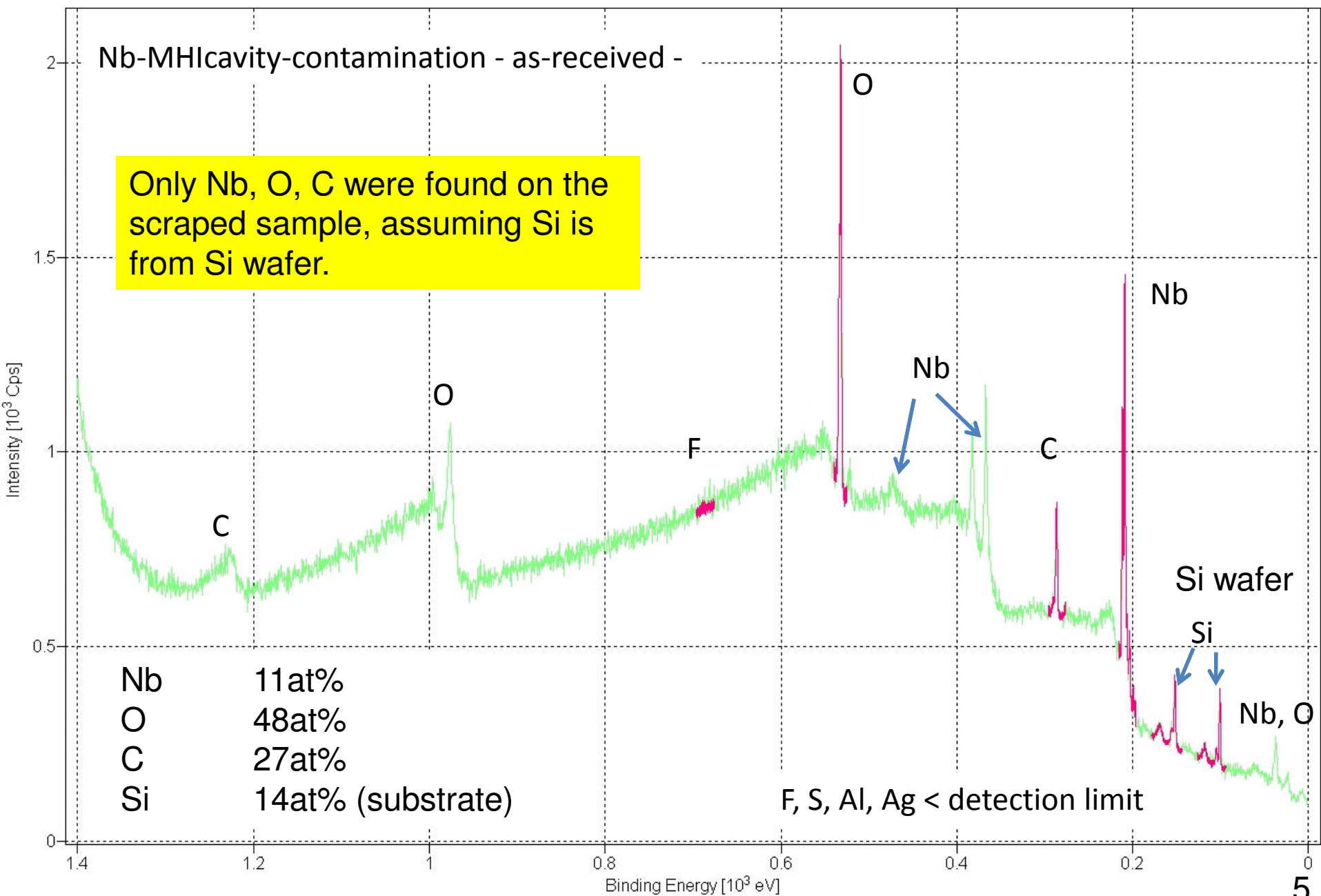
Sample preparation

Sample was set on Si wafer by silver paste.



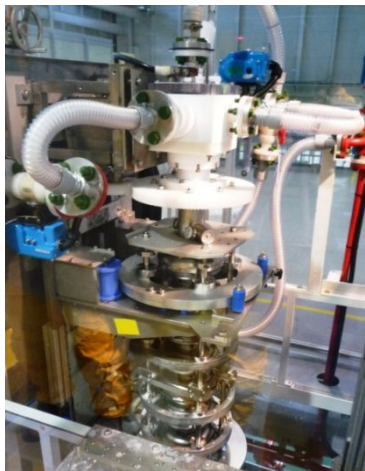
Scraped Nb sample

XPS analysis of scraped sample from old MHI 9-cell cavity

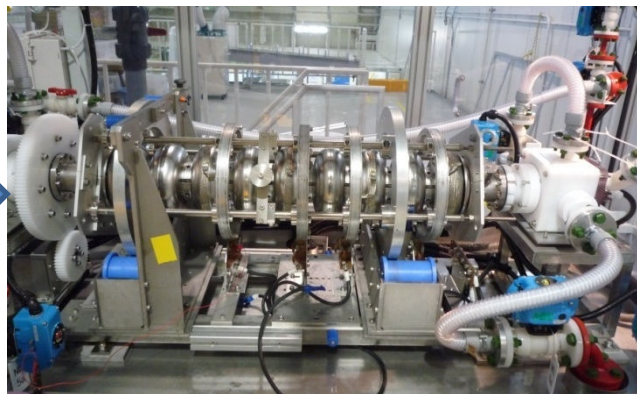
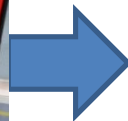


Draining process of EP acid

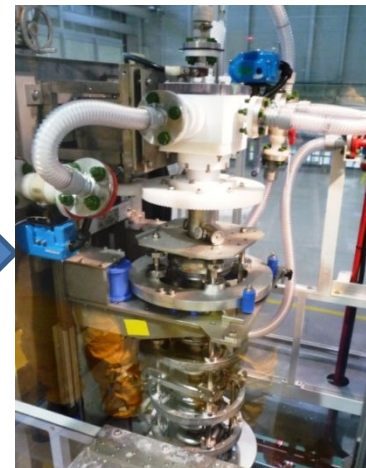
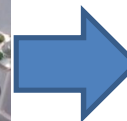
We are taking 30 min. to drain EP acid. This might be a reason for stain?



Drain EP acid for 15 min.



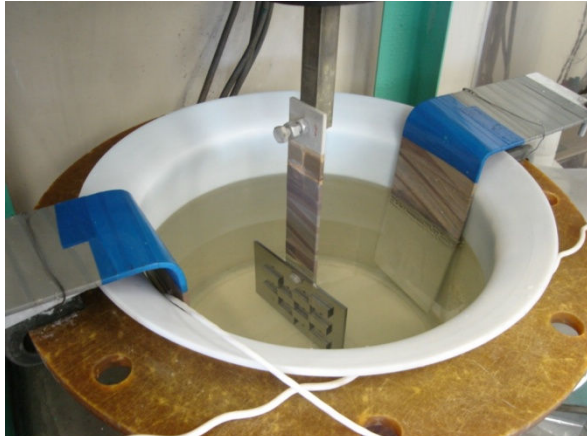
Horizontal for 5 min.



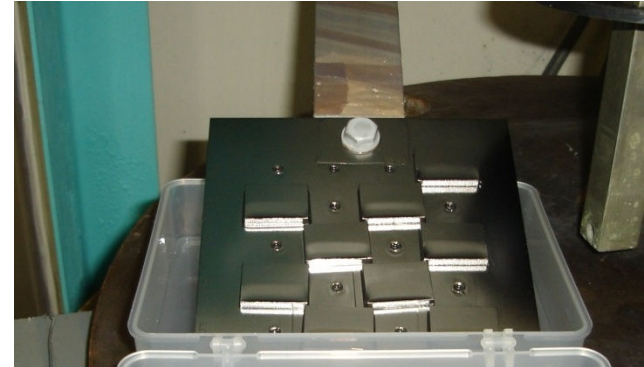
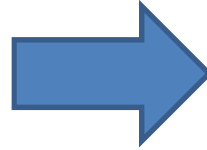
Drain EP acid for 10 min.
Then UPW rinse starts.

Labo-EP of samples at Nomura

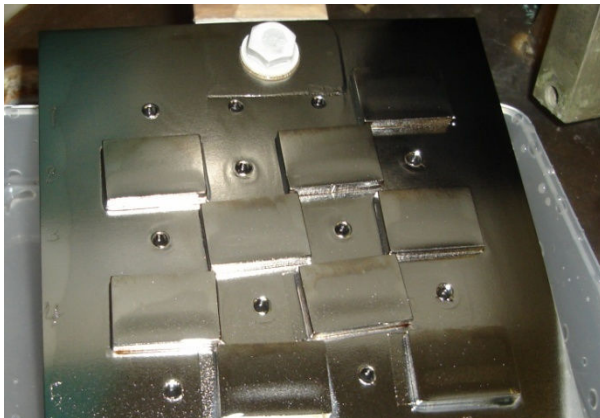
11 Aug. 2009



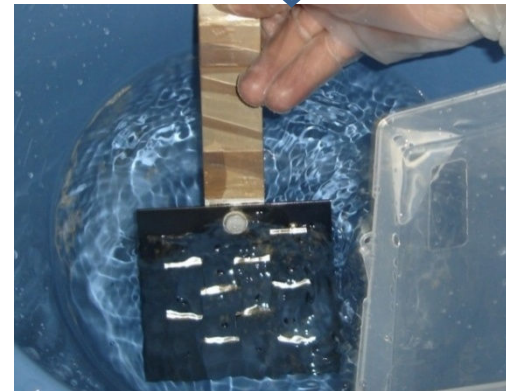
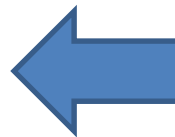
Labo-EP (20 μm) with **new EP acid** ($[\text{Nb}] = 0 \sim 0.4 \text{ g/L}$) at Nomura



Exposing the samples to the air for 70 min. w/o Pure-Water (P.W.) rinse. **No stain appeared.**

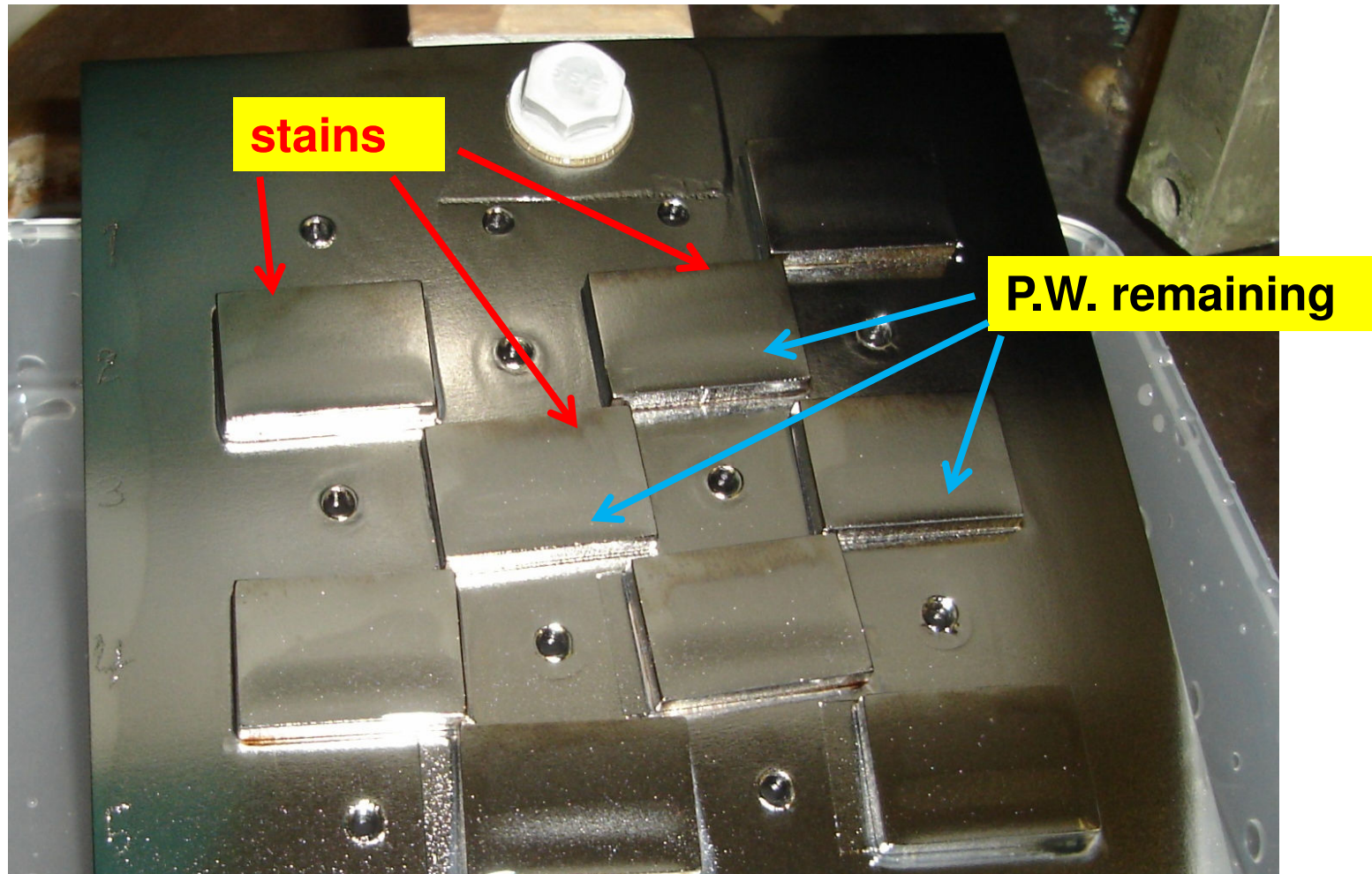


Stains appeared within a minute after exposing the samples to the air.



Light P.W. rinse for a few 10's seconds.

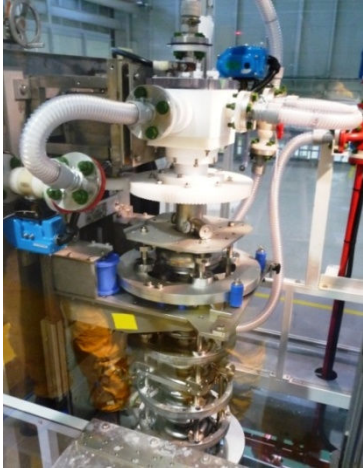
Exposing samples to the air after P.W. rinse



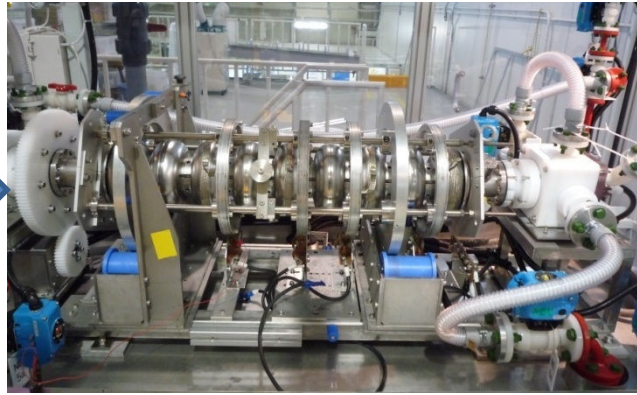
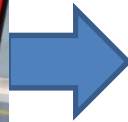
Stains appeared within a minute after exposing the samples to the air.
The stains appeared in the area where P.W. dried. (EP acid of [Nb] = 0.4 g/L)

Modification of first U.P.W. rinse process

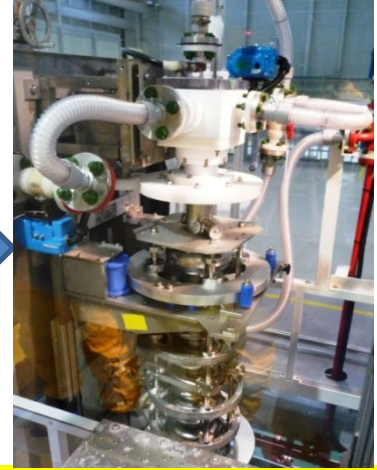
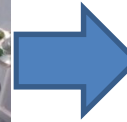
First U.P.W. rinse duration after EP-acid-draining at STF/KEK was extended.



Drain EP acid for 15 min.



Horizontal for 5 min.



Drain EP acid for 10 min.
Then UPW rinse starts.

The first U.P.W. rinse was extended **with overflow** for **a longer time**.

Original sequence: [pouring U.P.W. for 7 min. + draining for 5 min.] x 5

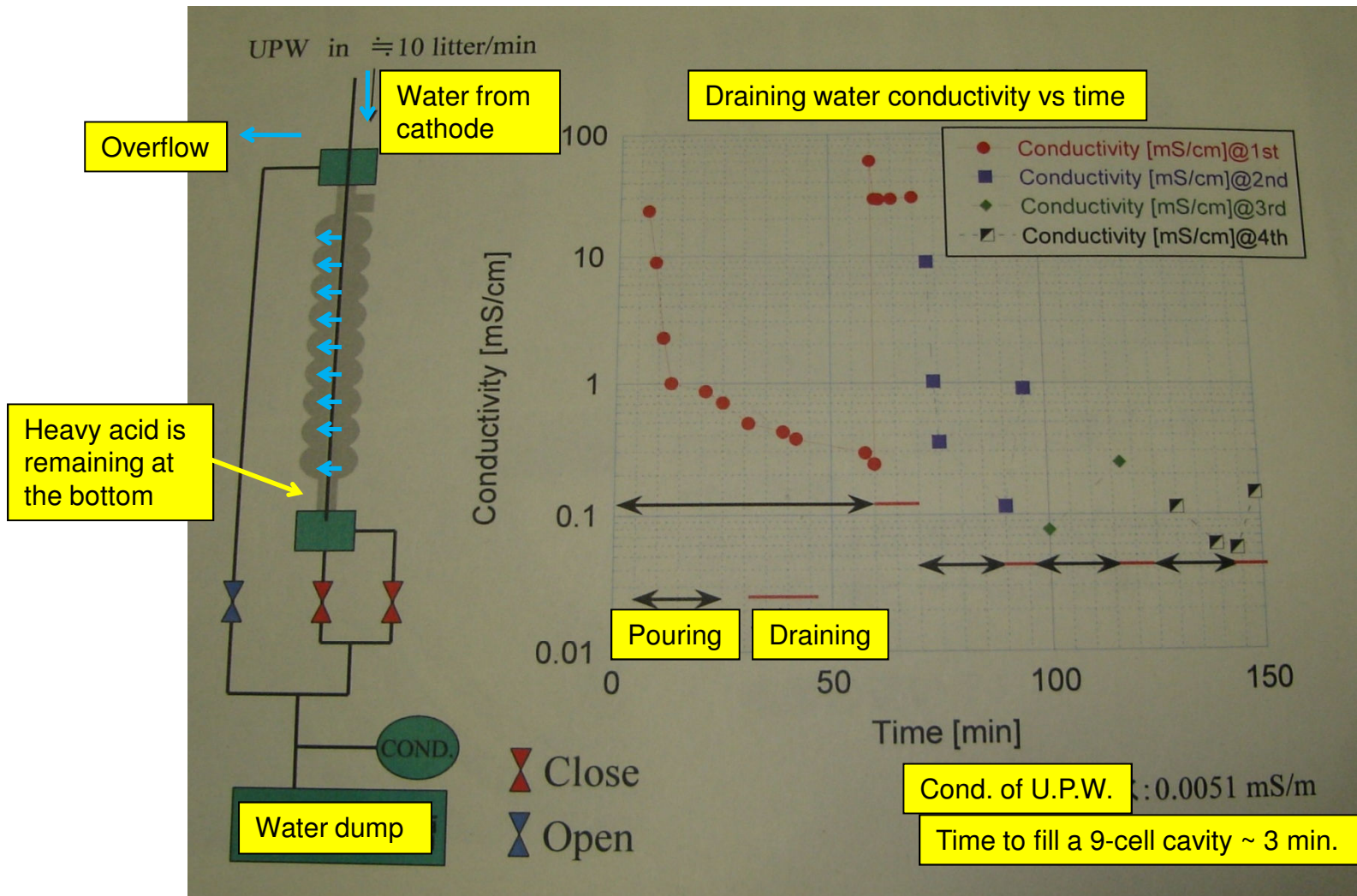
Modified sequence: [pouring U.P.W. for 60 min. + draining for 10 min.] + [pouring U.P.W. for 20 min. + draining for 7 min.] x 3.



MHI#9 (9-cell cavity) was processed with this modified sequence and reached $E_{acc} = 27$ MV/m at $Q_0 = 9 \times 10^9$ (quench), even with some field emission. More details about the results, see **"KEK cavity preparation for S1G" on 30th Sept. by E. Kako (KEK)**.

Still problem in the draining process of EP acid at STF/KEK

Still some stains are existing around the bottom cell (test cavity MHI#0).

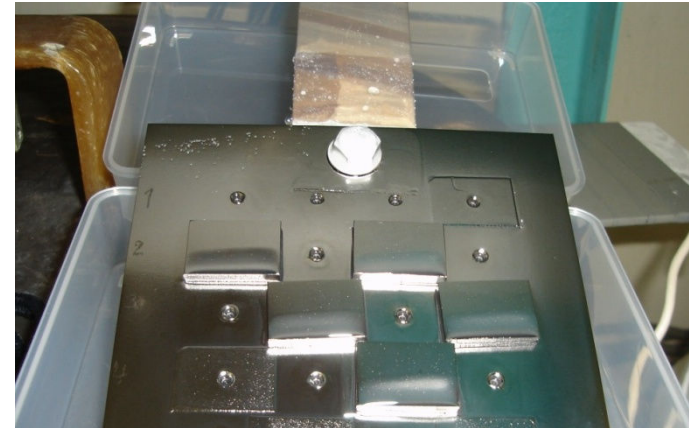
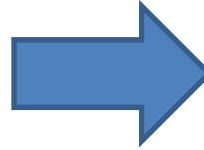


Labo-EP of samples at Nomura

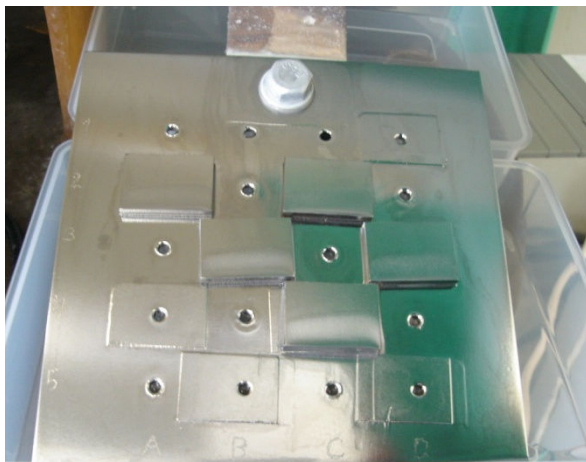
8 Sept. 2009



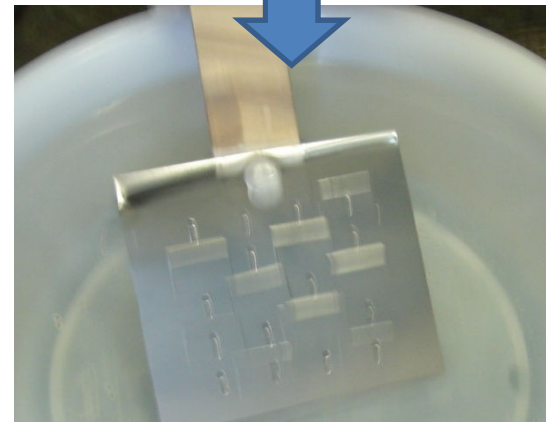
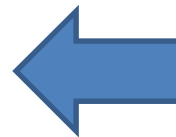
Labo-EP (20 μm) with **EP acid**
([Nb] = 4.4 ~ 4.8 g/L) at Nomura



Exposing the samples to the air for
30 min. w/o Pure-Water (P.W.) rinse.
No stain appeared.

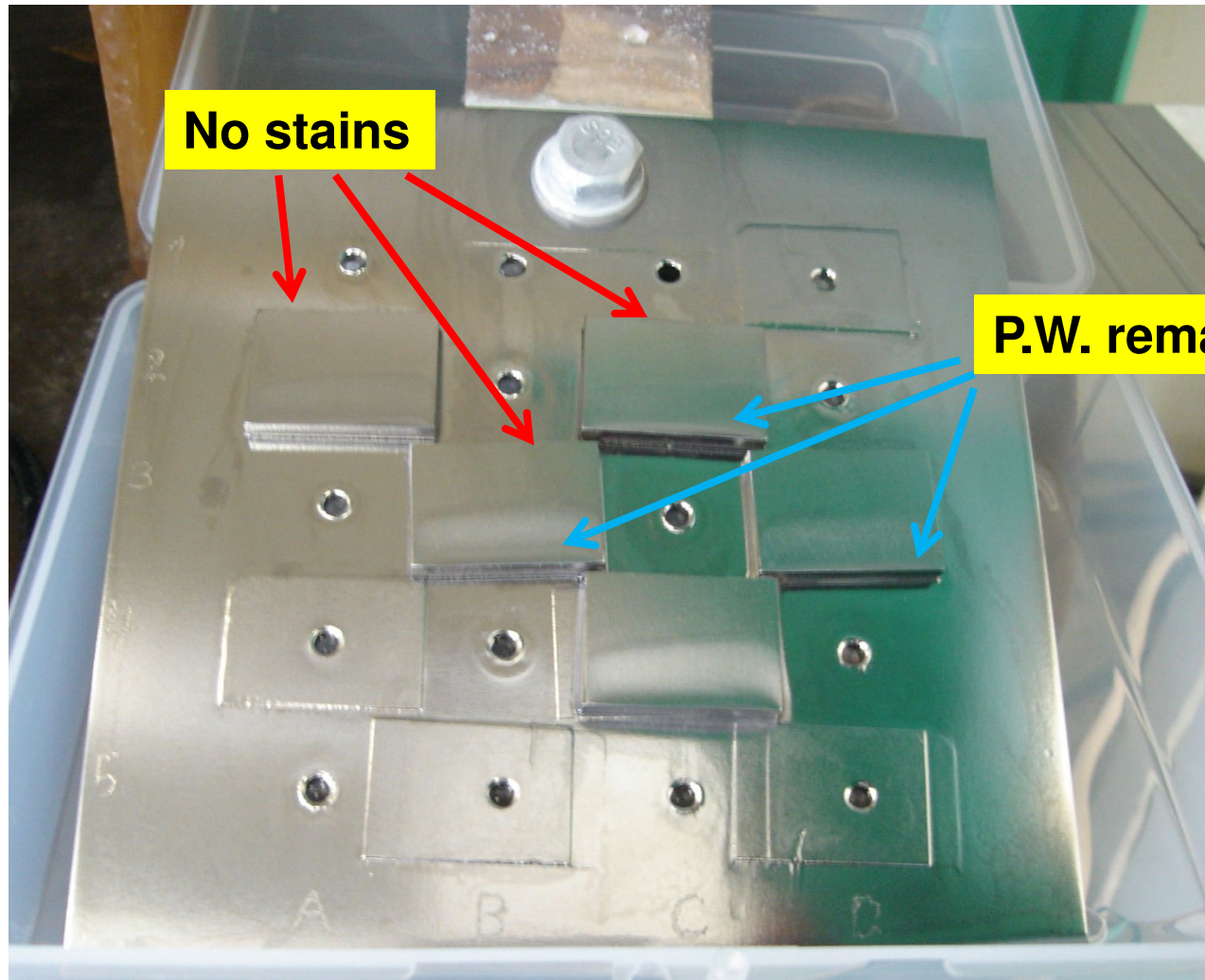


No stains appeared after exposing the
samples to the air.



Light P.W. rinse for a few 10's seconds.

Exposing samples to the air after P.W. rinse



No stain appeared after exposing the samples to the air.
(EP acid of [Nb] = 4.8 g/L at the end)

Summary of sample tests

[Nb] of EP acid (at the end of EP)	Duration of exposure to the air. Stains?	Duration of light P.W. rinse	Duration of exposure to the air. Stains?
0.4 g/L	70 min. No stain	A few 10's sec.	Stains appeared within a min.
4.8 g/L	30 min. No stain	A few 10's sec.	6 min. No stains
8.6 g/L	30 min. No stain	A few 10's sec.	4 min. No stains

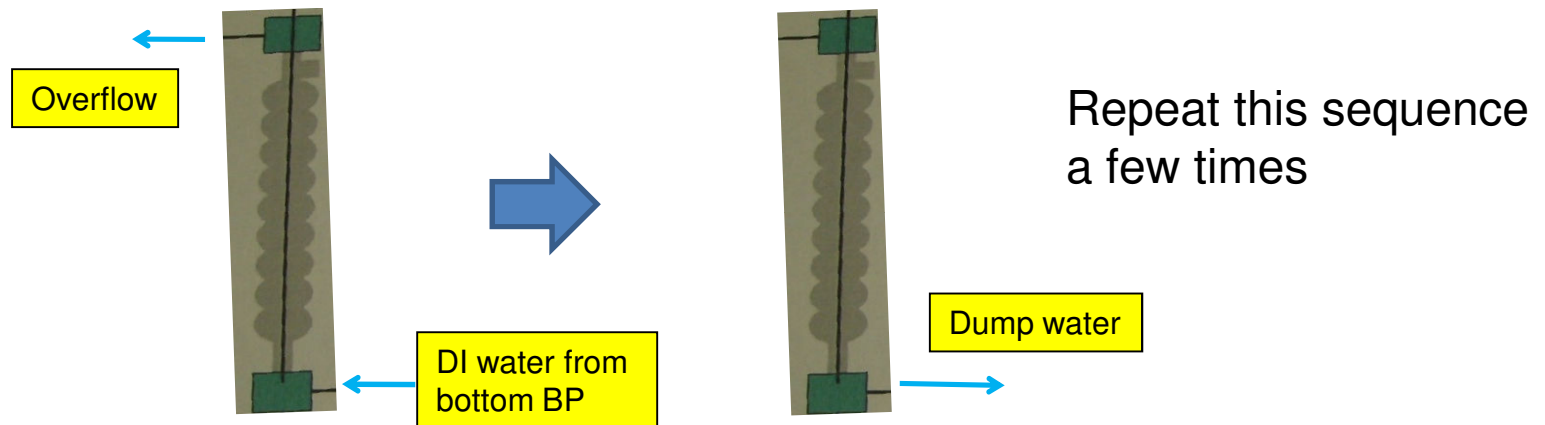
Considerations

- KEK EP-facility has a big EP-tank of 1000L. When we replace the EP acid with new one, we will keep using new EP acid with some 9-cell cavities for a while.
- Should we change the amount of EP-acid in the tank? Or should we develop a new U.P.W. rinse method, like introducing N2 during first U.P.W. rinse duration?

Comparison of EP process among laboratories

DESY and JLab have no stain problem.

- **DESY:** The final EP is done with the EP acid of [Nb] $\sim 10\text{g/L}$.
- **JLab:** New EP acid is used for the final (20 μm) EP 2 times, bulk ($>100\mu\text{m}$) EP, and then dumped. So new EP acid is used for the final EP is similar to KEK. But the rinse process is different from KEK.



Summary

- After we replaced the EP acid tank with new one, we encountered stain problem with MHI#6, #7, and #8.
- Sample scraped from test cavity was analyzed with XPS. Only Nb and O components were found.
- Stains were created only on the sample labo-EP'ed with new EP acid, and just after being rinsed and exposed to the air.
- Rinse sequence was modified at STF/KEK and Eacc of MHI#9 reached 27 MV/m. But still some stains were found in the test cavity around the bottom cell. Rinse sequence need more modification.
- DESY uses aged EP acid for final EP. JLab uses new EP acid for final EP but different rinse sequence. Both laboratories have no stain problem.