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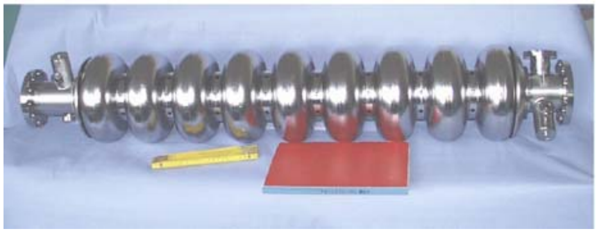

EP-Summaries

DRAFT April 11, 2007

D.Proch DESY, 30.03.2007

Summary documents of EP working groups at the TTC meetings Frascati

http://ilcagenda.cern.ch/conferenceDisplay.py?confId=desya0561&view=cdsagenda&showDate=all&showSession=all&detailLevel=contribution

and KEK
https://indico.desy.de/conferenceDisplay.py?confId=92

Content:

TTC Meeting WG#3 (KEK Japan)

R&D Efforts on Electropolishing Parameters
25-29 September 2006
Coveners: J. Mammosser, K. Saito, T. Tajima

Goals for this meeting:

The goals for this meeting were to review recent R&D efforts towards EP quality control, collect perspectives from industrial partners on the current process techniques and process quality assurance, review the progress on eliminating problem areas effecting performance, reviewing the current R&D results towards reducing performance spreads and to brain storm on process and system improvements.

- These goals were ambitious but were achieved by extending the meeting times and it is the convener's recommendation to increase the allotted meeting time in additional meeting on electropolish development to allow for increasing the depth of the discussions.

Electropolish Process Quality Control:

The leading concerns for the electropolish process are currently focused on the understanding of the final cavity surface conditions due to the nature of the electropolish process that leaves sulfur and other contaminants on the niobium surface and the ability to measure and control the quality of the electrolyte used for this process. During this meeting several presentations were given on electrolyte quality control. During this meeting the following methods of analyzing the standard EP electrolyte mixture (HF+ H_2SO_4) were introduced and discussed:

- Titration (Henkel Co.)
- Inductively coupled plasma optical emission spectroscopy (Henkel Co.)
- Ionic- chromatography (Henkel Co.)
- Total organic carbon (Henkel Co.)
- Nuclear magnetic resonance (Henkel Co.)
- Fourier transformation infrared spectroscopy- attenuated total reflection (Henkel Co.)
- Ion selective electrode method (FNAL)
- Capillary Electrophoresis method (Nomura Plating Co./KEK)
- Online Reference cells (DESY/JLAB)

Results from discussions on electrolyte QA:

1. There has been much activity in investigating what methods are available for electrolyte analysis with heavy input from industry (Nomura Plating and Henkel Co)
2. It is clear that no single method can fully analyze the electrolyte (mixture and content) and all methods are expensive, sensitive to errors and are difficult to interpret results. It is therefore extremely important to determine what aspect of these available methods can lead to better control of the process and improved cavity performance and reproducibility. These efforts are focused on the determination of how much HF and its associated ions, are in the electrolyte mixture at various stages of use and should remain a priority.
3. There is clearly not enough expertise and/or experience with these methods to implement any as an online QA method. Focus should be to continue using the industrial partners to develop more understanding of these methods, identify and choose key parameters to measure and use external resources (outside measurement labs) where possible to perform measurements until additional expertise is gained within the labs and in industry to determine if online monitoring is necessary and possible.
4. The inline IV reference cell implemented at DESY and JLAB is a less expensive approach to understanding how the electrolyte is changing over time. The DESY reference cell should be implemented at all electropolishing facilities to standardize measurement techniques, speedup understanding of how the process is changing and to be able to directly compare these facilities.
5. Analysis experiments were carried out by the Henkel Co with control mixtures using various analysis techniques. Two control mixtures were used both were diluted electrolyte solutions, one by adding DI water under low temperature controlled conditions (cool) and the second using city water with no additional temperature control (warm). The summary of results from these measurement are as follows:
    a. Slightly less fluorine concentration (F and FSO_3H) was measured with the warm than the cooled sample? One would have expected a stronger difference given the volatility of HF with temperature. ??? This test should be repeated to verify results.
    b. The warm sample had slightly more iron impurities in the solution and was most likely due to the difference in impurity content difference from the waters used to dilute the electrolyte. The other impurity concentrations seemed more consistent with the two mixtures.
6. Nomura Plating company/KEK performed several analysis experiments using capillary electrophoresis method. Here samples were procured to first develop calibration curves and then measurements were made on several electrolyte mixtures containing increasing amounts of Nb in solution as well as measuring HF concentrations of their production baths. The following are their results from these measurements:
    a. Measurements from control samples differed in results than that of the actual production baths for F^- concentrations. In control samples

the fluorine ion concentrations stayed constant during electropolishing up to 17 g/L of niobium generated in the process. The actual production baths however showed strong drops in fluorine ion concentrations with increasing levels of niobium concentration. This could be due to evaporation of HF from the baths due to the need for exhaust over the baths or removal of hydrogen gas during the processing.

The result on the control sample indicates that CE method can catches the total F^- ion in the EP electrolyte. As the chemical reaction equation in the EP process is well known, how much F^- was consumed is calculated. Therefore, how much active F^- ion still remained in the electrolyte is determined by the CP method. This is an important finding.

b. The electrophoresis method looks promising and additional studies needed for this method and should be supported and continued.

7. Additional methods such as Ion selective electrode analysis have been started but additional efforts are needed to understand the impact of this method.

Electropolishing R&D results

KEK has performed several single cell process studies towards a better understanding of performance spread using several process variations using tightly controlled and repeatable procedures. The following is the results from the discussions on these results:

1. In a pilot study with newly fabricated cavities a large spread in performance (39.1MV/m ± 8.2 MV/m) resulted from the first process cycle using standard KEK procedures. These procedures use CBP (100μm)as a baseline procedure to generate a uniform starting surface finish followed by a light BCP(10μm) to clean the cavity. Field emission onset was the main reason for the spread in performance.
2. An additional chemistry: EP(20μm)+EP(3,fresh acid) was performed on these cavities with a final chemistry with fresh acid on a closed cavity as the final processing followed by an additional HF rinse to remove chemical surface contamination. Results from this run showed a much improved performance curve (45.8MV/m ± 1.9MVm).
3. Additional CBP and similar chemistry ;EP(80)+EP(3, fresh acid) was performed resulting in a spread larger than the second test and smaller than the first process cycle with a 42.5 .0 MV/m ± 3.4 MV/m.
4. The difference of the spread between the first and the second could be understood by the contamination. The difference of the spread between the second and the third is interpreted that the heavy EP(80μm) could produce a persisting contamination.
5.
6.
7. The fresh acid on a closed cavity could make perfect sense that the surface should be cleaner with fresh acid than the typical process procedures. Additionally field emission is the cause for most of the spread and additional rinsing after chemistry seems to help produce less field emission.

8. The effects of the CBP are still unclear but it also makes sense that to get reproducible results starting with surfaces that are more uniform should lead to polishing that is more uniform and surfaces that are more reproducible.
9. The conveners recommend using CBP for further studies on nine cell cavities as a means to understand its impact in comparison with performance limits from the standard process and as a means to recover cavity performances from early quenches.

Results from Discussions on System and Process Improvements

During this meeting many times the discussions turned to what in this process should be changed to improve it in both method and hardware. The following is a summary of these discussions:

1. Most importantly all systems in current use have significant differences in both process used as well as hardware design. These differences are in some cases subtle and in other cases dramatic but all may have an effect in the end results for the process performance. Much of the discussions are aimed at flushing out these differences and trying to understand them.
2. One of the main areas of concern is the small volume of electrolyte used at DESY as well as JLAB. With increasing concern for precipitation of sulfur due to heating, excess niobium concentrations, contamination and loss of HF in the electrolyte, a small volume of electrolyte should make the process more sensitive to any of these problems. The result of these discussions is that all agreed that a larger volume of electrolyte would be better for consistent results and current system are difficult to change this feature but future designs should take this into consideration.
3. Current system designs have all produced good results and should be adequate to understand and improve the current process. Any major changes to the current process could reset the learning curve.
4. All agreed that constant current control should be implemented for S0 studies but first demonstrated on single cell test cycles to ensure compatibility then be scaled to nine cell cavities.

Progress since last TTC meeting

During this meeting, a careful review of the list generated for the possible causes for performance spread was performed and resulted in removing the following items that were listed:

Scatter resulting from subtleties of EP process?

1. Process parameters: non-reproducibility, non-uniformity of material removal, Set-up: acid level, cathode bag, cathode shielding, current leads, T-control
2. Reproducibility in acid composition (DESY), Draining and rinsing: overheating? for multi-cell cavities

Scatter caused by “environmental” problems?

3. Malfunction of system,
4. Vacuum problems,
5. Problems during testing

Reasons for removal of these items:

1. DESY has done such a great job at controlling and documenting each production process cycle that significant evidence exist to be able to remove these possible problems due to the tight control of their procedures and processes. Data from these procedures shows that they were reproducible and indicates that the spread in performance was not due to variations from these items listed but most likely some other variable that is not directly measured or controlled as of yet.

Items left on the list:

1. Problems during rinsing and/or assembly
2. Human errors
3. Q-disease: unpredictable material?

These items were left on the list due to no strong evidence supporting their removal. There are currently two spreads in performance that are forming the large distribution of performances, early field emission onset and early quenches. There is strong evidence that early field emission is mainly due to generic surface contamination and can be easily modified by additional cleaning steps.

After the electropolish process step the cavity surface is in its most contaminated state and there is concern that additional cleaning steps are not currently adequate to produce consistent results. The major concern is sulfur contamination which is non soluble and extremely difficult to remove and that the HPR cleaning may not be reproducible between labs or effective in covering all surfaces of the cavity interior.

Both of these topics are subjects of TTC studies currently and need to be addressed for S0/S1 efforts to be successful. Human errors are still problematic but results can be successfully identified and separated from study results. Q-disease is less of a problem today with very few cases during DESY production cycles and with none identified this year?

This is still not understood It has already been determined that the S0 testing shall include Q-disease tests until evidence supports removing this additional step from the testing process.

Additional topics discussed:

- Field emission must be solved at each lab due to the differences in facilities, system hardware and procedures in use, there can be no single cure for all facilities. What is important is to discuss and share results from investigations performed and to develop a better understanding of the differences of each system and procedures used. What would help this process would be to start implementing the same measurements at each location where possible, record the same parameters and openly share data from processes and cavity test results, there is already good progress in this area.
- Large grain single cell cavity tests have shown excellent results even without EP and more studies should be carried out to determine the spread in performance from this material in comparison with small grain structures.

Recommendations to TTC Board

1. DESY needs to document evidence to remove said items from the Frascati list (Reschke)

2. TTC board needs to manage efforts on reducing field emission (Coordination of resources and direction)
3. DESY must provide EP I/V cell design to be implemented at other labs, this is the easiest way to compare system performance (Matheisen)
4. Labs working on tight loop must identify an electrolyte QA program and provide details and an implementation schedule by end of this year
   KEK (Saito), Jlab (Mammosser), DESY (Matheisen)
5. Need to complete EP operational specifications by end of this year, (Tajima)
6. At next TTC meeting:
   1. DESY will provide analysis of the next 30 RF tests (Matheisen)
   2. JLAb will provide analysis of all tight loop RF tests (Mammosser)
   3. KEK will provide analysis of all tight loop RF tests (Furuta)
   4. JLab will implement and provide field emission data from witness samples from tight loop (Mammosser)
7. TTC board in collaboration with ILC S0/S1 group must determine what and how data is going to be managed for this R&D effort
8. Saito (KEK) will coordinate efforts towards resolving correct method for removal of sulfur and be implemented by end of March 07
9. TTC board should review or assign task to review field emission reduction plans at each lab
10. Labs must provide their detailed plans for reduction in their performance spread by the end of this year

Proposal for an R&D Plan towards better Understanding of the Electropolishing of Niobium Cavities

P. Kneisel, K. Saito, D. Reschke

Jan. 17, 2006

During the last year issues concerning the electropolishing of niobium cavities have been discussed at various meetings such as the TTC meeting at DESY in March 2005, the ILC Snowmass workshop, the SMTF workshop at FNAL in October 2005 and now at the TTC meeting in Frascati.

A summary report about Electropolishing activities worldwide will be published in the near future [1]

It has become very clear that the major problems have to do with contamination of the electropolished surfaces as well as with unpredictable hydrogen dissolution, resulting in some cases in “Q-disease”. Better “on line” monitoring of the process seems to be a desirable QA/QC activity.

A. Contamination and Rinsing studies

Already in 1971, when the electropolishing process was developed at Siemens AG in Germany [2], it was recognized that the electropolished surfaces were contaminated with residue from the polishing bath mixture of hydrofluoric acid (10 parts) and concentrated Sulfuric acid (85 parts) and that rinsing of the surfaces was a very important step in the surface preparation. At Siemens rinsing in a diluted solution of hydrogen peroxide was as well applied as a subsequent step/several steps of anodizing in ammonium-hydroxide solution and stripping the pentoxide in hydrofluoric acid under ultrasonic agitation (oxipolishing) The rinsing in peroxide solution had the effect of fully oxidizing reaction compounds on the surface to soluble chemical products.

Electropolished surfaces were investigated with XPS by Grunder [3] at the Kernforschunszentrum Karlsruhe and he found – besides various states of oxidation – sulfur in the form of sulfate and fluorine F^- .The sulfur contamination, which seems to be obvious by the odor of the surfaces (even after evacuation and exposure and after high temperature heat treatment), could be totally eliminated by by oxipolishing, whereas boiling the samples in water reduced the sulfur contamination by a factor of 10 and converted the sulfate state to a sulfide state. The fluorine contamination could not totally be removed by either treatment, but was significantly reduced.

During the TRISTAN R&D and production phase K.Saito [4] developed – starting from the Siemens process – "continuous horizontal" electropolishing, which is the presently used technique for multi-cell elliptical cavities in most laboratories (exceptions are Cornell University, where vertical electropolishing is developed and at Argonne National Lab, where the Siemens intermittent electropolishing is applied). During the mass production, sulfur contamination of the bath was encountered at a rate of 4 mg/l of solution for a 80 micron material removal on a 5-cell TRISTAN cavity. It was promoted by the buffing abrasive contamination in the EP acid, which was applied to remove mechanically surface defects prior to the heavy EP (80 micron). Investigations showed, that the precipitation of sulfur in the bath could be reduced by rinsing the system with CCL_4, CS_2 or acetone or by filtration with activated carbon. Consequently, KEK added a pre-EP (3micron) process before the heavy EP (80micron) in order to remove the contaminated surface layer from the buffing. The acid used for pre-EP was only used for this purpose. The TRISTAN cavities were treated in a batch of four cavities. EP acid was replaced every four cavities EP (80micron). The TRISTAN cavities apparently did not suffer from sulfur surface preparation, because KEK used new EP acid for the final EP (10micron) after the EP acid contamination problem and rinsed in peroxide solution. However, they observed a soft MP(2-point 1st order) barrier around Eacc~7MV/m in every cavity, which might still indicate a contamination problem.

Sulfur contamination in the electropolishing system at DESY has been reported since quite some time [5] and it is suspected that sulfur deposits on the surface are in some cases responsible for early onset of field emission at fields $\leq$ 25 MV/m. This suspicion is based on a comparison of repaired cavities from cryomodule 3, which were prepared by BCP in the same time period as the EP cavities and did not show field emission, excluding problems with the HPR system or during assembly. Sulfur can be dissolved in alcohol as initial tests have shown.

In the context of the CARE/JRA1 in work package 5.1, carried out at Saclay, optimization studies on electropolishing solutions and parameters are undertaken. In these studies it has also been found that sulfur is a contaminant on the polished surfaces and in the polishing system [6]. The generation of sulfur is inherent to the polishing mixture and the process.

- *Studies of surface contamination of EP surfaces with appropriate surface analytical methods are highly desirable and by applying different rinsing procedures one should be able to re-confirm the results from 3 decades ago*.
- *In parallel, rinsing studies on single cell cavities should be carried out to optimize cavity performance.*

As a reasonable procedure one can employ a reference cavity with a baseline performance, which will be in subsequent steps always electropolished by a chosen amount, eg. 10 micron, and rinsed differently. By carrying out at least 3 experiments per rinsing procedure, one should get some limited statitistics.

An alternative procedure could be to use several different cavities for each rinsing activity. However, one has to bring each of these cavities to high performance, possibly multiplying the amount of effort going into this activity.

B. “Q – disease”

In 1991 it was discovered [7] that cavities made from high purity niobium can suffer from unexpected losses of several orders of magnitude, if they are exposed to temperatures between 150K and 75 K for longer periods of time. This increase in losses was in subsequent investigations at several laboratories identified as caused by precipitation of hydrogen in the form of the α-hydride; it happens, when interstitially dissolved hydrogen in the niobium matrix can cluster into this hydride. The interstitially dissolved hydrogen enters into the metal during the chemical surface treatment of the niobium either by BCP or electropolishing. (Is this true? During chemical processing, doesn’t generated hydrogen as a byproduct of the chemical reaction enter into the bulk Nb??) Remedies against the uptake of hydrogen/Q-disease are a surface treatment at low temperature (<15 C of the acid bath) in the case of BCP, shielding of the cathode with a cathode bag and possibly adding a small amount of nitric acid [8] to the acid mixture in the case of electropolishing. In general, hydrogen can be degassed in UHV at temperatures $\geq$ 600C (hydrogen degassing procedures applied to RRR niobium range from 600C for ~ 10 hrs to 800 C for 3 hrs) , preventing Q-disease; also, a rapid cooldown of the cavity through the dangerous temperature region prevents the precipitation of residual hydrogen into hydrides. However, this procedure might not be applicable for large cryo-systems such as an ILC cryomodule. Therefore it is important to eliminate the possibility of additional losses caused by this phenomenon; a degassing heat treatment as has been applied e.g. to the cavities during the TRISTAN construction, would contribute to additional costs in the cavity preparation.

It has been reported [9] that in some cases Q-degradations were encountered unpredictably on 9-cell TESLA type cavities, even though the electropolishing procedures had been applied identical to cavities, which did not suffer from degradation.

At KEK, where presently a very successful single cell program is being pursued with a modified surface treatment procedure consisting of centrifugal barrel polishing, heat treatment and electropolishing, no Q-disease has been identified. However, prior to this outstanding results, cavity performances were very mixed and only after the appropriate electropolishing conditions were re-established at the electropolishing shop have these excellent results been obtained [10]

It seems to be very important to understand the conditions, under which Q-disease is encountered. For this to happen, the following R&D activities are proposed:

- *<u>Each test of an electropolished cavity should be tested for Q-disease,</u>* meaning, that after an initial test, which might not show a Q-degradation because of fast cooldown, the cavity should be held for ~ 12 hrs at a temperature around 120C and then re-cooled again.

  Such a test sequence will give a baseline for the frequency of Q-degradations due to precipitation of hydrogen.
- Another possible source of non-reproducibility could be encountered during the rinsing procedure of the cavities at the end of an electropolishing cycle. The cavity surface is covered with an acid film (and possibly there is quite a bit of acid trapped in piping and fixturing volumes, which cannot be drained very well), which will react exothermically with the rinsing water and possibly raise the surface temperature. It is known from experiments with bcp [11] that a surface temperature above ~ 30 C increases the amount of hydrogen pick-up by the niobium. Single cell cavities, which usually do not show Q-disease (after fast cooldown) are easier to handle and might be less vulnerable to this potential source of hydrogen pick-up.

  In addition, the niobium, when covered with a thin acid film containing hydrofluoric acid, has no protective oxide layer, which can prevent the pick-up of hydrogen. This situation is the same at the beginning of an EP cycle, where EP acid is put into the cavity without any voltage potential, which can prevent the hydrogen-ion from entering into the niobium material.

It is recommended that a series of tests be made, which explore these possible sources of hydrogen dissolution in the niobium.

- The electropolishing process as applied presently is done at an acid temperature of $\geq$ 30C, a temperature, which for the bcp process is significantly too high. The hydrogen emanating from the process is being shielded from the non-exposed part of the cavity (the part sticking out of the acid), however there is hydrogen generated in the acid also. If the distance of the cathode to the anode (cavity) is insufficient, the hydrogen stream in the acid can reach the niobium surface and possibly can be dissolved in the material. This should not happen, if the set-up is optimized and the hydrogen is prevented from reaching the niobium surface, which is either inside the acid bath or outside (the top part). As reported in [10], the appropriate adjustment of the cathode distance and hydrogen screen is a very important parameter for successful electropolishing conditions.

  It is recommended that a series of electropolishing tests (most efficiently with single cell cavities) with modified cathode and cathode screen distances is undertaken and the cavities are checked for Q-disease

  Hopefully, the result of these tests will be an optimized electropolishing configuration, which does not lead to hydrogen pick-up by the niobium surfaces.

C).” On-line” monitoring and control

It has been experienced that the material removal on a typical cavity surface is non-uniform. Even though the aluminum cathode is shielded at the irises and the major voltage drop supposedly happens close to the anode, there is nearly a factor of 2 difference in material removal between iris and equator of a cavity (the smaller amount is removed at the equator). Whether this difference is due to different polarization curves in the two cavity regions or due to temperature gradients or due to different acid flow patterns is not clear. It has , however, been established recently [12] that a shaped cathode leads to a more uniform polarization curve over the entire surface and should result in a more uniform material removal. Besides an experimental program, which can explore such improved conditions with cavity tests, DESY has recently acquired a computer simulation program from the University of Bruxelles, which can be used to simulate different polishing conditions and configurations as well as flow patterns.

During the electropolishing process the concentration of hydrofluoric acid, which is essential for good polishing conditions, is reduced and the polishing conditions are changing. For maintaining reproducible polishing conditions it seems to be important to implement in any EP system an "on-line " monitoring of HF concentration and of the polarization curve.

- *It is recommended to explore to its fullest the simulation program (this is happening already at DESY)*
- *It is recommended to implement in any existing EP system "on – line" monitoring and data logging of polarization curves and HF concentrations*
- *It is recommended to investigate with an experimental program the causes for the non-uniform material removal by e.g. modifying the temperature distribution, the acid flow pattern, the cathode shape, the cathode shielding or the location and number of current leads.*

D). Acid Composition

It has been reported at the TTC meeting in Frascati that at least one delivery of pre-mixed acid (" Honeywell Mixture", 10 parts HF, 85 parts H_2SO_4) to DESY in summer of 2005 had an unusual composition [9] and behaved differently during the electropolishing. It has been "hypothesized", that exposure of the mixture to hot weather during the delivery caused the modifications.

DESY is pursuing the industrialization of the electropolishing process with the company Henkel, mainly with single cell cavity preparations so far. Henkel is mixing the EP solution "in house" with nominally the same ratio of acids as in the "Honeywell mixture", however, the company reports a much more "active" mixture.

- *It is recommended that the subject of acid composition and different behaviour of nominally the same mixtures will be addressed; most likely, this can be done by chemical analysis and comparison of polarization curves measured on samples under identical conditions.*

The table below summarizes the proposed R&D activities:

Problem	Proposed Activity	Priority
Contamination Field Emission	Rinsing studies with samples (XPS,SIMS…) Rinsing studies with single cell cavities	1
Non-reproducible appearance of Q-disease	Test any electropolished cavity for Q-disease Can overheating during initial rinsing cause Q-disease? Optimizing studies for cathode/screening geometry	1 2 2
Monitoring and control	Implementation of “on line” monitoring and data logging of polarization curves and HF concentrations Exploitation of EP simulation program Investigation of the cause for non-uniform material removal	1 1 2
Acid composition/ decomposition	Chemical analysis of acid mixture (nominally equal) Polarization curves on samples	2

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Summary of electropolishing discussions at Frascati TTC meeting on 5-7 December 2005 and at smtf meeting at fnal on 5-7 october 2005

Tsuyoshi Tajima* for the Working Group attendees

Abstract

This document presents an outcome of the discussions at the TTC meeting at Frascati on 5-7 December 2005, which was a continuation from the SMTF meeting held at FNAL on 5-7 October 2005. Our goal was to identify the cause of the results spread of EPed 9-cell cavities that have been tested mostly at DESY. While the spread might not have been caused only by the EP itself, the fact that the spread is larger than BCPed cavities may suggest that the EP process or EP-related contamination due to such as sulfur may be the cause of the problem. After the discussions on EP parameters and current issues, we suggest that the following be carried out with R&D efforts as highest priority items: 1) further study how important it is to control HF content and what is an appropriate range, 2) establish the best way to eliminate sulfur, a reaction product while EP and is insoluble to water, and 3) study effectiveness of pre-polishing such as CBP at KEK. In addition, we encourage other ongoing R&Ds including vertical EP studies as supporting elements and future cost reduction. WG1 was led by P. Kneisel, K. Saito and D. Reschke at the TTC meeting and all the presentations can be found at http://www.lnf.infn.it/conference/ilc05/programme.html. The SMTF meeting at FNAL was led by T. Tajima and C. Boffo and the presented files are shown at http://ilc-dms.fnal.gov/Workgroups/SMTF/Collaboration Meetings/SMTF Collaboration Meeting Oct 5,6,7 2005/.

ACRONYMS

ANL	Argonne National Laboratory
BCP	Buffered Chemical Polishing
CBP	Centrifugal Barrel Polishing
CEA	Atomic Energy Comission, France.
DESY	Deutsch Electron Synchrotorn Laboratory
EP	Electro-Polishing
GMR	Giant Magneto Resistance 2nd order gradiometer
HPR	High Pressure (water) Rinsing
HRC-EP	Horizontally Rotated Continuous Electro Polishing
INFN	National Institute of Nuclear Physics, Italy
INFN/LNL	INFN Legnaro
INFN/Mi	INFN Milano
KEK	High Energy Accelerator Research Organization, Japan.
QA	Quality Assurance
SMTF	Superconducting Module Test Facility
TTC	TESLA Technology Collaboration
WG	Working Group

INTRODUCTION

The HRC-EP that was developed by KEK and Nomura Plating company [1] has been adopted by DESY and JLAB. DESY has carried out 90 EP processes since it started in 2004 for a total

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processing time of 199 hours [21]. JLAB has processed a few 700-MHz, 805-MHz and 1.5-GHz multi-cell cavities with their system and has identified some issues to improve their system, although due to funding shortage they have not been able to do a lot on EP in the recent years.

The major goal of this WG was to discuss EP parameters and the reason why the spread of EPed 9-cell cavities is so large, i.e., $20<E_{acc}(MV/m)<35$ as shown in Fig. 1 based on mostly DESY data [21]. We discussed important parameters, what are the issues that need further R&D and need to be clarified or solved.

There were some presentations on single/large grain cavities and seamless cavities, but they were excluded from this document to focus on EP.

BRIEF HISTORY OF EP

Peter Kneisel gave a comprehensive review of the history of EP that has been adopted for treating accelerator structures [3]. The following shows the brief history.

- In 1971, H. Diepers and coworkers of Siemens AG developed a new method of electropolishing Nb within a government funded collaboration agreement with the Kernforschungszentrum Karlsruhe (KfK).
- The process was subsequently used for the surface treatment of cavities (RF separator, helix, R&D) at KfK.
- In ~1975, it was "exported" to HEPL [5], Cornell and KEK.
- In ~1980, K. Saito and Nomura Plating company modified and developed the HRC-EP method for 508 MHz 5-cell TRISTAN cavities [1]. The EP related figures in Refs. [1] and [8] are shown in Appendix A.

Appendix B shows chemical reactions that can occur during EP.

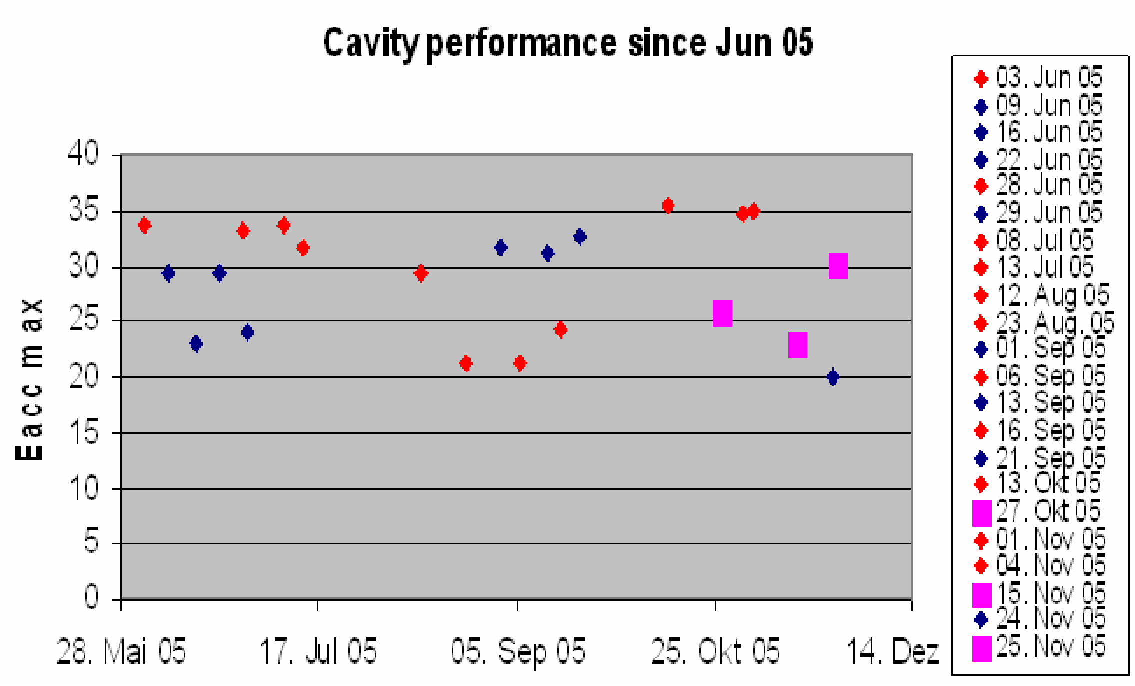

Figure 1: Performance of EPed DESY 9-cell cavities since June 2005 [21]. Red diamonds show the results after 120 °C baking and the purple squares show those after fast cool down of the cavities that have Q disease.

INSTITUTES THAT HAVE EP CAPABILITY

Table 1 shows a list of institutions and their experience. Presently, DESY and KEK/Nomura have the full capability of electropolishing 9-cell cavities. JLAB has a facility that is large enough to accommodate a 9-cell cavity, but they have no experience so far. Appendix C shows the EP facilities at DESY, JLAB and KEK/Nomura. Appendix D shows small facilities for shorter cavities or for sample tests.

Table 1: List of institutions that have EP capabilities.

Institution	**Facility**	**Experience**	**Ref.**
ANL	for low-β	Since late	[14]

Institution	Facility	Experience	Ref.
	structures	1960s	
CERN	1-cell	Since 2001	??
Cornell	Vertical EP	Since 2003	[10]
DESY	Sample tests	Since 2001	
DESY	HRC-EP	Since 2003	[6,7]
FNAL	Small tests	Since 2004??	[13]
JLAB	HRC-EP	Since 2003??	[9]
INFN/LNL	R&D on GMR and Automated EP	Since 2004	[2]
KEK	HRC-EP	Since ~1980??	[1,8]
Saclay	Small tests & mono-cell	Since 2004??	[11,12]

IMPORTANT PARAMETERS

The parameters in Table 2 have been agreed to be important and most of them have been the same as or scaled from the data described in K. Saito's paper [1]. These ranges were mostly determined by sample roughness and brightness measurements.

Table 2: Important EP Parameters.

Parameter	Range	Notes
Current density	30-100 mA/cm^2	
Voltage	8 – 16 V	depending on the current density and HF content
Bath temperature	25 – 35 °C	
HF concentration	60 – 90 cc/L	
Rotational speed	<1.8 rpm??	scaled from 0.7 rpm for 508 MHz
Acid flow rate	?? l/min	scaled from 60 l/min for 508 MHz cavities

Another important fact is that the viscous layer on the anode (Nb) surface needs to be preserved for EP to occur, i.e., too much turbulence that breaks this layer will lead to an unsuccessful EP.

MATERIALS

Some materials have been found incompatible with the EP acid. They are:

- Viton: There are various types of Viton and there might be compatible Viton.
- PVDF: DESY has used this for the part that needs welding since this is the only material that seemed compatible and weldable.

Teflon® or polytetrafluoroethylene (PTFE) is the ideal material to be used wherever possible.

Other issues related to materials that need to be reminded are:

- The cathode needs to be made of a pure aluminum (1100) to avoid corrosion.
- The tubes for heat exchanger could be pure aluminum, but the EP acids need to be mixed elsewhere before adding to the heat exchanger to avoid corrosion of Al tubes. (ANL uses Al tubes.)

NEW RESULTS AND OTHER ISSUES

Recent studies with 9 cm^2 samples at Saclay supported by EU CARE program have shown the following results [12].

- After 5-7 g/L Nb has been reached, the surface state starts to degrade. This, however, seems to be at least partially due to a decrease of HF content and could be different for actual cavities.
- The increase in HF content increases polishing rate, Nb solubility and acid solution lifetime, and it decreases the production of sulfur. The drawbacks, however, are corrosion of Al cathode, safety issues and difficulty in temperature control due to high removal rate.

DESY has found the following issues.

- The acid mixture delivered from a company was not the right mixture in the past, suggesting that the QA of delivered acid is an important issue.
- Some EPed cavities show Q disease, but not all the cavities, which may suggest that there were some difference(s) during EP process, although they could not find any difference in recorded operating parameters.
- There seems to be difference in removal rates in different cells according to the field flatness measurements. KEK has not had this problem, although they have checked with only one 9-cell cavity so far. This could have been an effect of handling after the EP [22].

POSSIBLE CAUSES OF EPeD CAVITIES RESULTS SPREAD

The question whether it is due to EP itself or to the subsequent process remains to be answered. However, assuming that the subsequent process has been the same quality as BCP cavities, the following are the possible causes that have been discussed so far.

- The EP parameters have not always been controlled to be within the range shown in Table 2.
- Removal of chemical contaminations such as sulfur was not always complete.
- The quality of the delivered acid mixture was not always the same.
- The initial cavity surfaces before EP were not the same quality among different cavities.

R&D ITEMS

Table 3 shows the currently ongoing R&D efforts at various laboratories. Through the discussions at Frascati and FNAL, we have identified the areas where we need to intensify R&D in a timely manner. They are:

- Effect of controlling HF content and determine an appropriate range for proper EP.
- Effect of eliminating sulfur with either oxipolishing, H_2O_2 rinse or methanol rinse, etc.
- Effect of pre-polishing such as CBP on the results spread. Currently, KEK is the only place where an advanced CBP facility is available and hopefully they can do this R&D expeditiously.

Table 3: Ongoing R&D's on EP at various institutions.

Institution	Topics
Cornell	Vertical EP
DESY	Parameter validataion with 1-cell (with Henkel), components optimization with 9-cell cavities
FNAL	Papameter study with small samples
INFN/LNL, INFM/Naples	Automated EP
U. Naples [2]	Magnetometry for better cathode configuration
JLAB	Parameter validation, Continuous monitor and addition of HF
KEK/Nomura	Effect of CBP on the EP reproducibility
Saclay	Parameter validation, solution aging, method of surface qualification
U. Bruxelles	Analyses of EP phenomena??
U. Wuppertal	Surface roughness measurement with Laser

COORDINATION OF EP R&D

While there are regional programs such as CARE that include EP studies, establishing a more global coordination is desirable to conduct more focused R&D in a timely manner. The following are suggestions that might expedite the R&D.

- Initiate the global R&D effort soon.
- Open a web page specialized on EP R&D
- Make an e-mailing list and circulate eLetters as frequently as possible to disseminate new results for discussion.

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[17] K. Saito, private communication.

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APPENDIX A: DATA FROM K. SAITO'S PAPERS [1, 8]

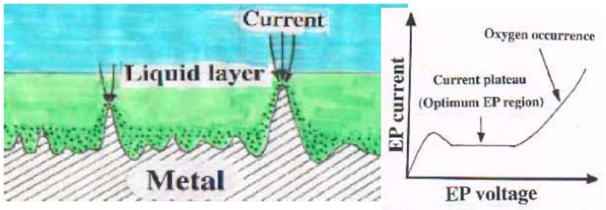


Figure A.1: Schematic representations of leveling effect by EP (left) and typical I/V curve during EP (right).

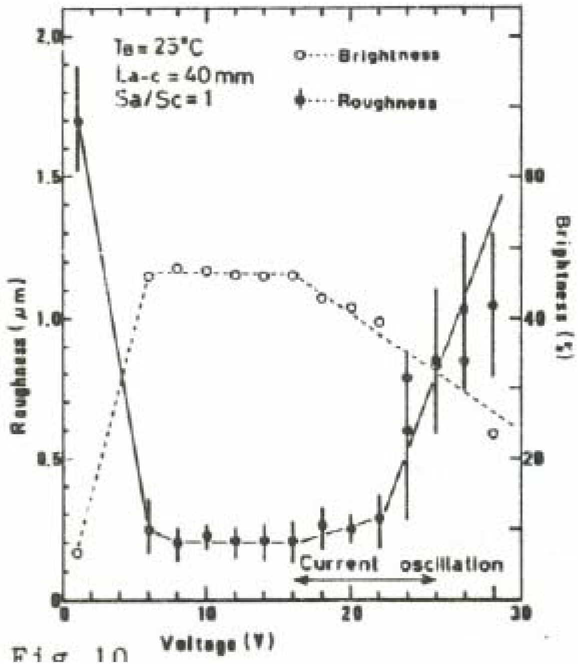


Figure A.2: Roughness and brightness as a function of voltage.

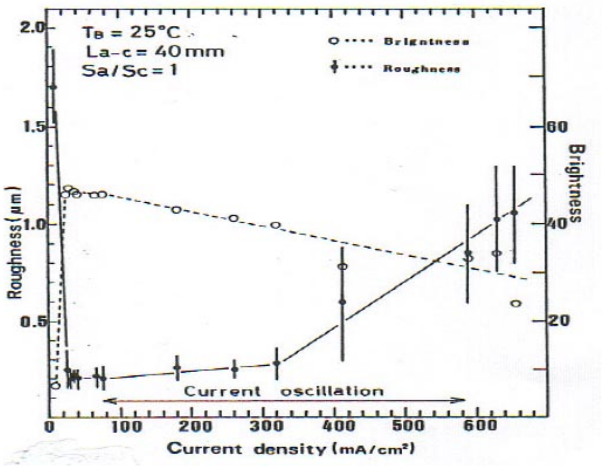


Figure A.3: Roughness and brightness as a function of current density.

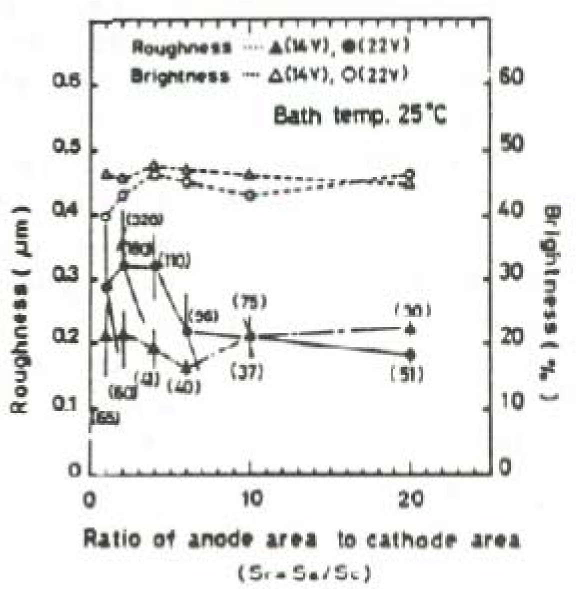


Figure A.4: Roughness and brightness as a function of ratio of anode to cathode surfaces.

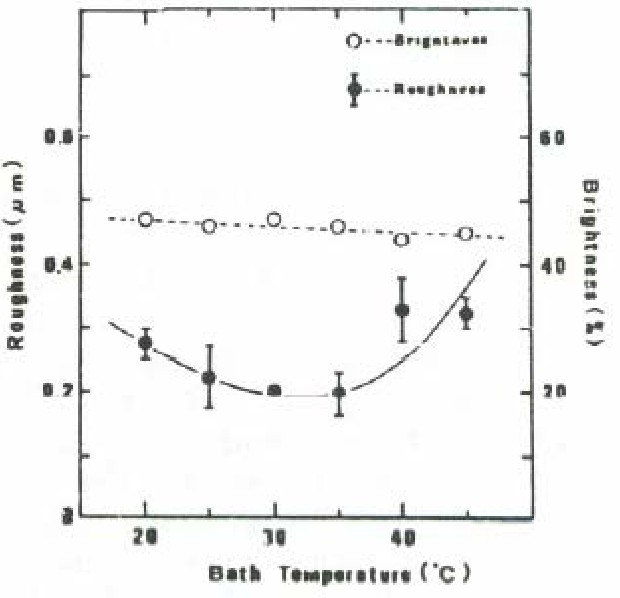


Figure A.5: Roughness and brightness as a function of bath temperature.

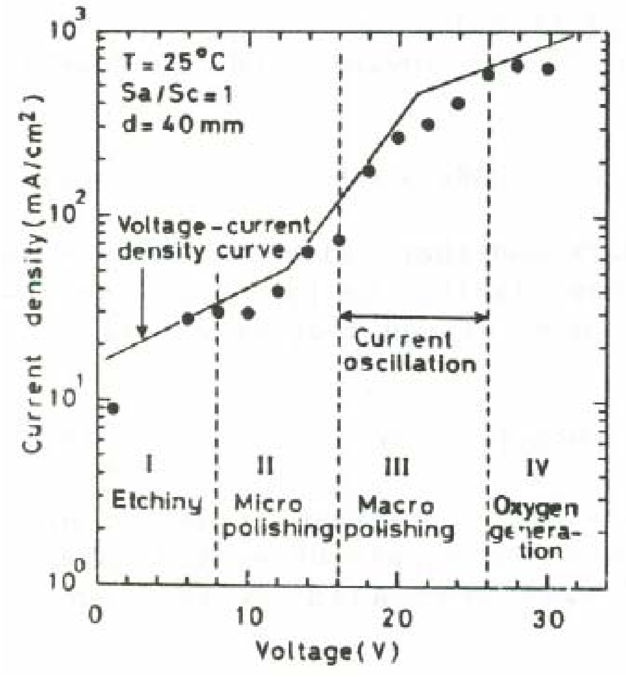


Figure A.6: Current density vs. voltage and various surface states resulting from these conditions.

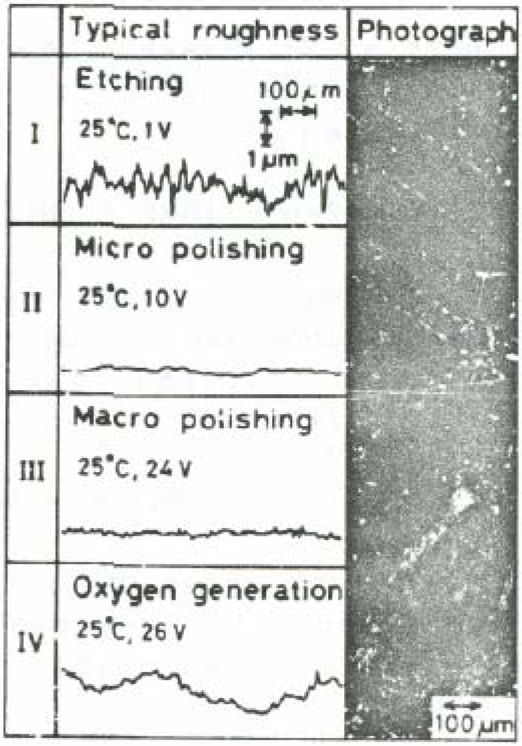


Figure A.7: Typical surface roughness and photograph for each polishing reason.

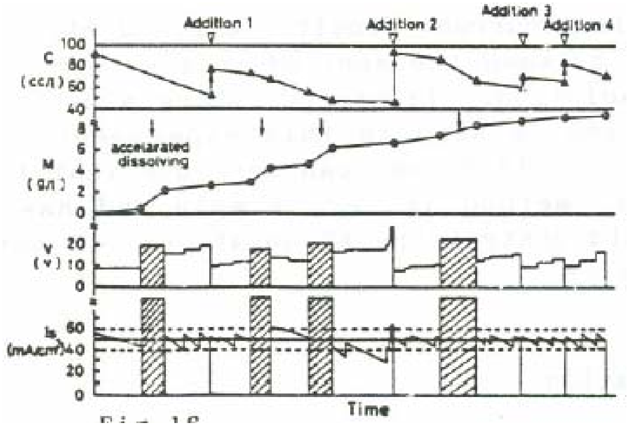


Figure A.8: Sample test on a refreshing method of an EP solution by the addition of $HS0_3F$.

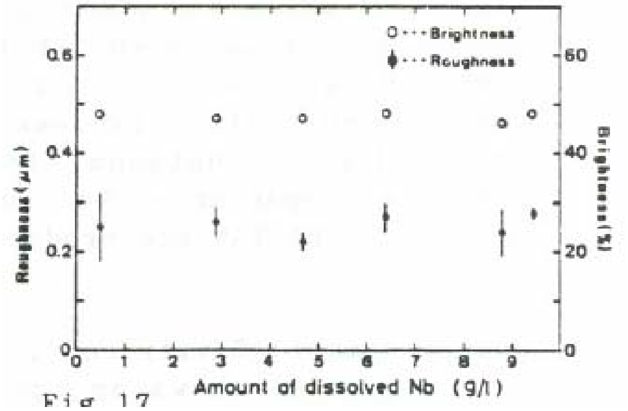


Figure A.9: Roughness and brightness as a function of the amount of dissolved Nb.

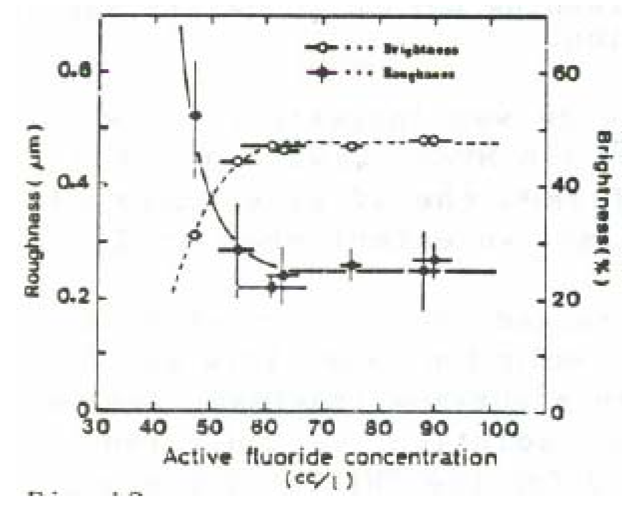


Figure A.10: Roughness and brightness as a function of HF concentration.

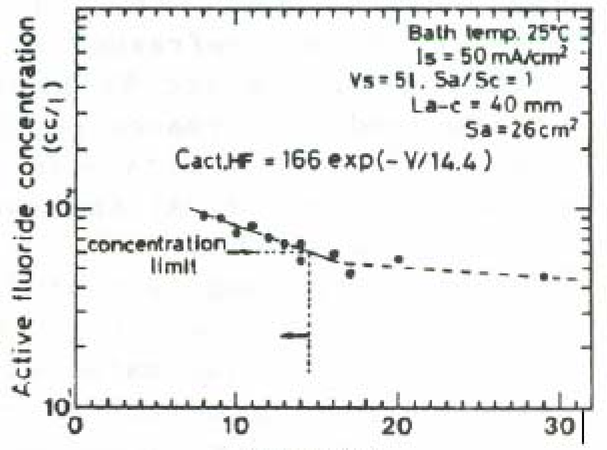


Fig. 19

Figure A.11: HF content as a function of voltage to keep the current density at 50 mA/cm^2.

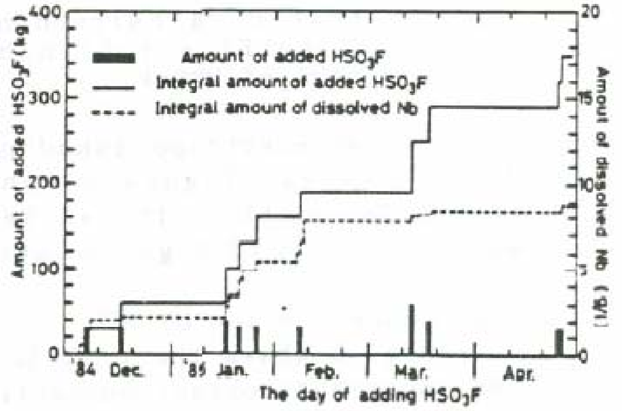


Figure A.12: Effect of adding HSO$_3$F on the amount of dissolved Nb.

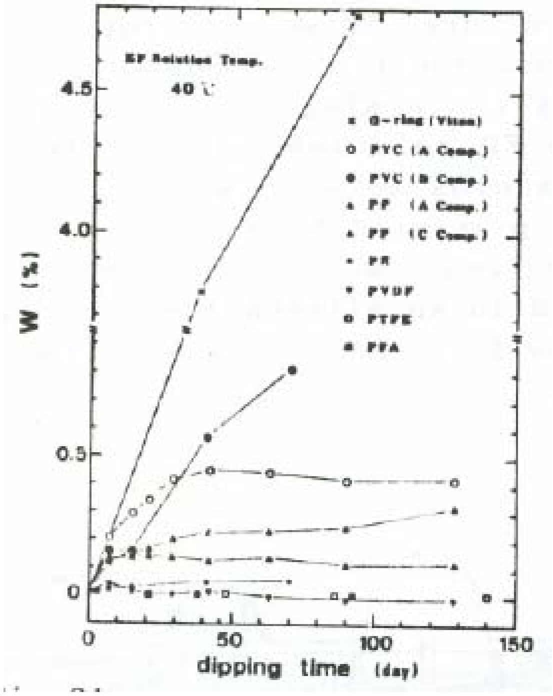


Figure A.13: Change of weight as an indication of absorption of EP solution for various materials.

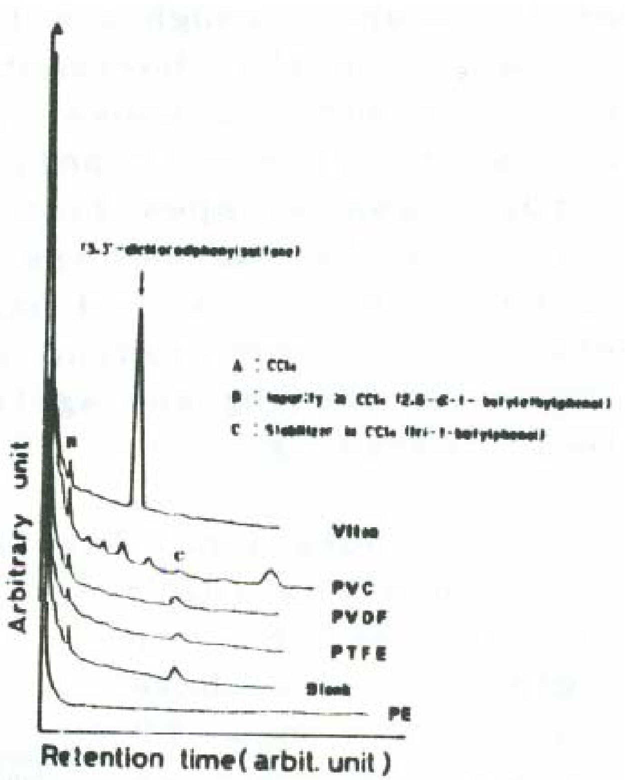


Figure A.14: Dissolving of plastic materials and Viton into an EP solution for two months soaking.

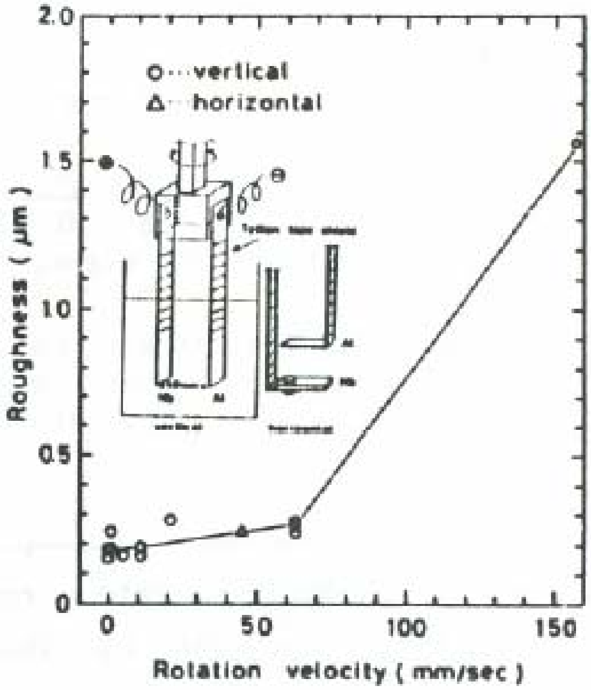


Figure A.15: Roughness as a function of the anode speed relative to the EP solution.

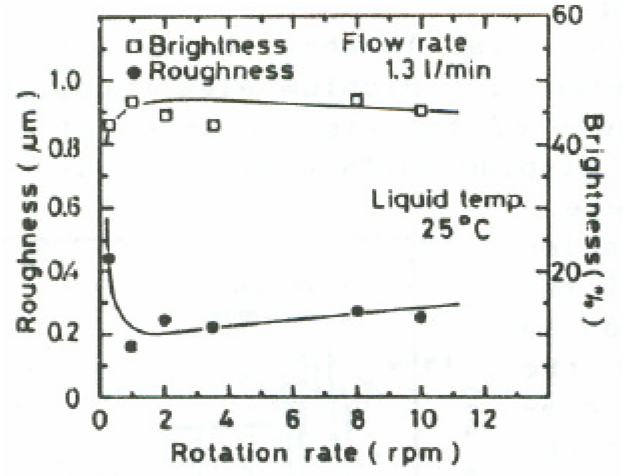


Figure A.16: Roughness and brightness as a function of cavity rotation speed. The cavity was a 1.5 GHz single cell.

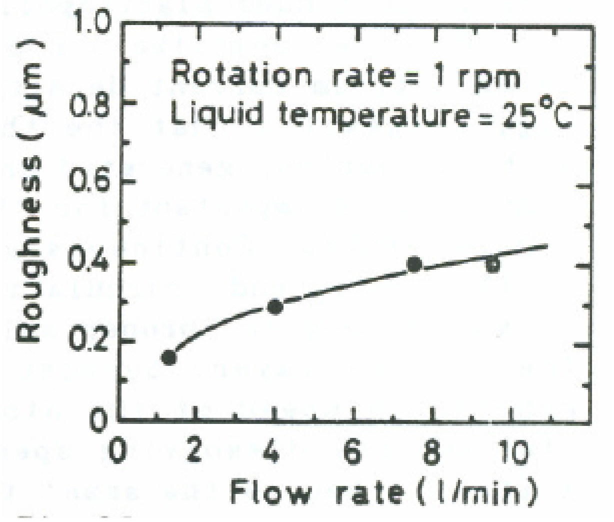


Figure A.17: Roughness vs. flow rate at a rotation speed of 1 rpm. One 1.5 GHz single-cell cavity was used.

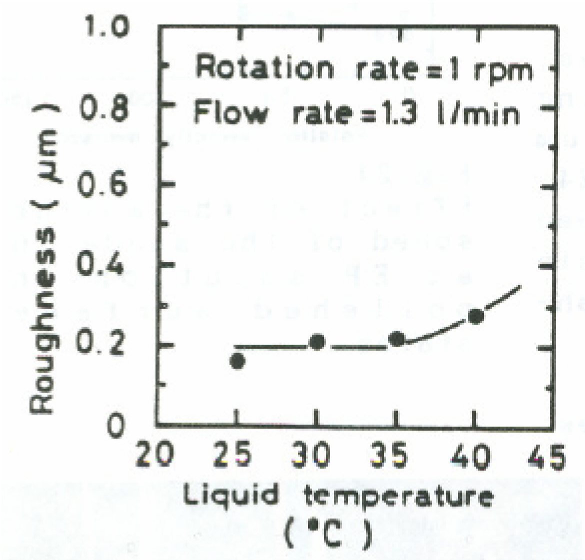


Figure A.18: Roughness as a function of EP solution temperature at a rotation 1 rpm and a flow rate of 1.3 l/min.

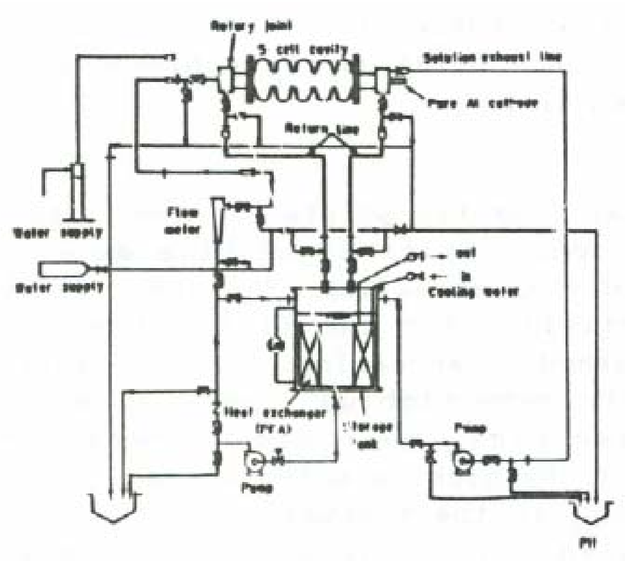


Figure A.19: Flow chart of the EP system for the TRISTAN 508 MHz 5-cell cavities.

APPENDIX B: CHEMICAL REACTIONS THAT OCCUR DURING EP

$$
\begin{aligned}
2\mathrm{Nb} + 5\mathrm{SO}_4^{--} + 5\mathrm{H_2O} &\rightarrow \mathrm{Nb_2O_5} + 10\mathrm{H}^+ + 5\mathrm{SO}_4^{--} + 10e^- \\
\mathrm{Nb_2O_5} + 6\mathrm{HF} &\rightarrow \mathrm{H_2NbOF_5} + \mathrm{NbO_2}\cdot 0.5\mathrm{H_2O} + 1.5\mathrm{H_2O} \\
\mathrm{NbO_2}\cdot 0.5\mathrm{H_2O} + 4\mathrm{HF} &\rightarrow \mathrm{H_2NbF_5} + 1.5\mathrm{H_2O}
\end{aligned}
$$

(B.1) [19]

$$2Nb + 10HF + 2H_2O \Rightarrow 2H_2NbOF_5 + 5H_2$$

(B.2) [??]

$$Nb_2O_5 + 10HF \Rightarrow 2H_2NbOF_5 + 3H_2O$$

(B.3) [15]

Cathode corrosion (Al) and Sulphur deposition

$$2Al + 4H_2SO_4 \rightarrow Al_2(SO_4)_3 + S\downarrow + 4H_2O$$

solution → alcohol rinse

(B. 4) [15]

APPENDIX C: 9-CELL CAVITY EP MACHINES IN THE WORLD

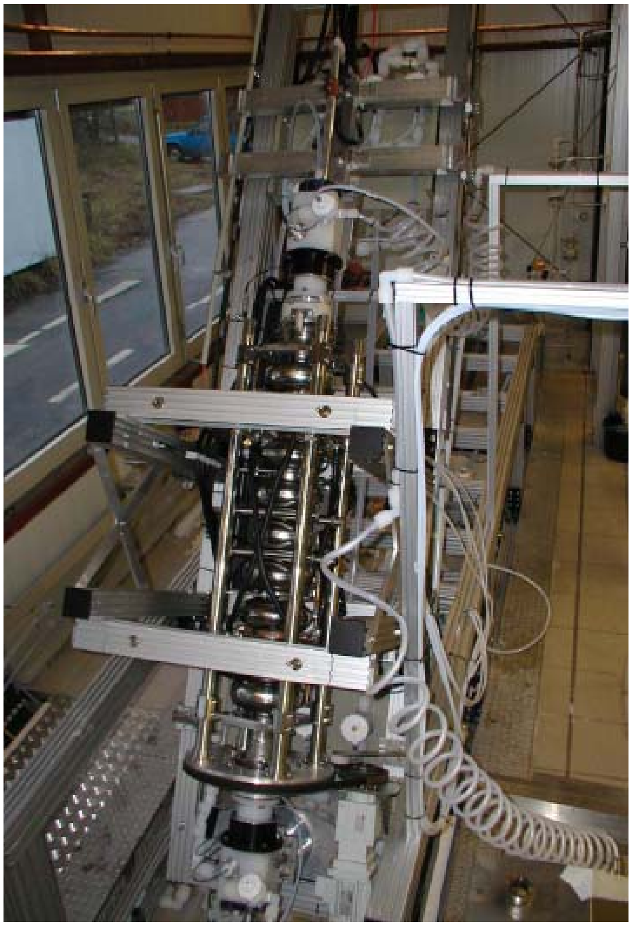


Figure C.1: DESY in Germany. [16]

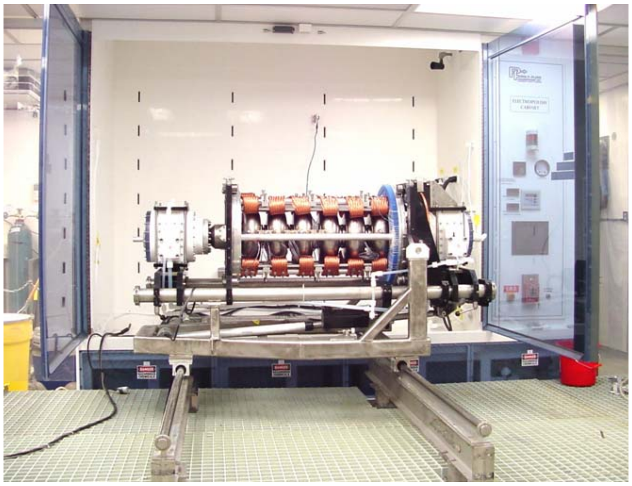


Figure C.2: JLAB in the U.S.A. [18]

??

Figure C.3: KEK/Nomura in Japan. [17]

APPENDIX E: SINGLE-CELL AND SMALL EP MACHINES

Figure D.1: Henkel company in Germany. [16]

Figure D.2: CEA/Saclay in France. [2].

Chemical surface treatment process

		units	12/5/05 Decision	DESY	Jlab	KEK (Nomura)	KEK (STF)	FNAL SMTF	ANL	Cornell	DESY Henkel
Electropolishing				EP 1/9	EP 1/5	EP	EP 1/9				
	Situation			Horiz	Horiz	Horiz	Horiz		filled	vertical	Horiz
	Control			const voltage		const current	continuous				
	Voltage	V		17?		15-25	const current		15 20		(12-18)
	mode			continuous	continuous	continuous	15-25		pulsed		continuous
	Current	mA / cm^2		49-58		40~50	40-50		30 to 50		
	Power	W / cm^2		1							
	Temperature of liquid (start-end)	C		24-35		25~32	25-32		28 32		22-30
	Temperature of cavity	C				30~35	30-35				
	Acid flow rate	l/min		9-11		6	6		agitate		(7.5)
	rotation speed	rpm		1.5-2	1	1,4	1,4				(0.5)
	H2 screen details			360 degrees	180 degrees	180 degrees	180 degrees				none
	nitrogen purge for H2			30lpm	1cfm	none	none				
	cathode masking			iris/BT	iris/BT	iris/BT	BT				
	seal material			PVDF		viton	viton				
	Tolerable Nb content	gpl		10		9	9				
	Heat exchanger			PVDF tube	PVDF tube	PFA tube	PFA tube		3003 series AL		
	Acid quality (grade)			electronic	electronic	reagent	reagent		reagent		

	storage volume	l		150	240	1000/100	1000				800
	masking material			PTFE tape	PTFE tape	PFA film	PFA film				
	cathode to NB ratio										
	Cathode material	AL purity		100		1100 series	1100 series				
	Cathode shape			tube	tube	tube	tube				shaped
	Cathode dia			1.0 inch	1.3inchs	25 mm	25,0				
	etching speed	micron / min		0,4		0,4	0,4				
	total removal	micron		180-200		80	80		150		
	additional removal	micron		?		30	30				
	total time for a treatment	hr		6+2 h		3.5	3.5				
	contact location			2 stif ring, 2 dish head		all EQ plus beam tube	all EQ plus beam tube				
	experience			> 200hr		> 3600 hr	0				
	diff temp in to out	C		8		10					
Care after EP											
	water rinsing	min		60	60	60 *	60				
	drying	min		0	30	0	0				
	Time till HPR	min		120	30	30~45	30~45				
	water temp of rinse	c		20	29	50C only for 9cell case	50C only for 9cell case				
	conductivity water out	uS		400		use UPW	use UPW				
	resistivity water out	Mohm-cm									
Chemical polishing				BCP 1:1:2	BCP 1:1:2	BCP 1:1:1	NA				
	Etching speed	micron / min		1,1-0,9	1.2-0.7	8~10 **					
	Acid Temperature	C		15-18	10	Room temp					
	Soak time	min				1~2					
	Acid Flow rate	l/m		3-20	15,2	stirring					
	Drain time	sec		80		20 ***					
	Purpose					pre-cleaning					

High Pressure Rinsing for Assembly										
		units	12/5/2005 Decision	tol	DESY	Jlab	KEK Nomura	KEK AR-east	KEK STF	FNAL (A0
Basic parameters										
	Pressure after final filter	bar	70-120		100	96	80 ***	70	70	83
	Period	hr/cell				0,83	1	1	1	0,125
	Flow rate	liter / min			15-20	9,1	10	10	10	18
	rotation/up-down		separate motion				separate	separate	separate	
	Cavity Rotation speed	rpm				2	15	15	15	10
	Wand up down/speed	cm/min				1	22	22	22	3,5
Water quality										
	Resistivity	MΩ cm			18,2	18,2	18	6	18	18,2
	TOC	ppb			<1	< 6	~15	~150	10	<20
	Bacteria count	cfu				< 50	<1	~10	< 1	<20/liter
	meas. Point					POU	POU	POU	POU	in line
	Particle count at pressure	counts/ml				0,88	Non	Non	Non	NA
	Nitrogen overflow				yes	cleanroom air	no	no	no	yes
Spray Head Nozzle										
	nozzle material				**saphire**	Ti	ss303	ss303	ss303	Sapphire
	head material				SS 1.4429	ss316L	ss303	ss303	ss303	SS316L
	number of holes				8	2	8	8	8	8, 2 types
	hole size	Dia (mm)			0,4	0,9	0,6	0,6	0,6	4-0.4,4-0.2

	jet direction	angle in degrees			+30 up -30 down	40	-45, 0, +45	-45, 0, +45	-45, 0, +45	
	type of nozzle				solid jet	fan jet	solid jet	solid jet	solid jet	
	errosion				no	?	Yes **	Yes **	Yes **	No
	measurement needed for all									
Filter										
	filter size	um			0,02	0,04	0,22	0,22	0,22	0,04
	location				POU	POU	POU	POU	POU	POU
Other cares										
	Cavity Environment				Class 100 (10)	Class 100 air brought in	Class 1000 air brought in	Class 1000 air brought in	Class 100 air brought in	Class 100 N
	dust free mechanism					?				N2 purge, roc vacuum
	potential difference between nozzle and cavity				under investigation	Unknown	10~20mV		10~20mV	
	grounded?				yes	no	No	No	No	
	particle migration free					?	In/Out isolation	In/Out isolation	In/Out isolation	
	Water Temperature	C			20	29	room temp	room temp	room temp	
Transportation										
	transfer from hpr to drying area	min			1	10	240	60	60	
	Transfer time from chem to HPR (EP)	min			45	60	90	90		
	Transfer time from chem to HPR (BCP)	min			3	30	60	30		3 hrs
	Transfer method (EP)				water filled	wet	wet	wet	wet	
	Transfer method				water filled	wet	water filled	water filled	water filled	Wet/filled

	(BCP)									
	sealing after chem for transfer to hpr				closed	closed	closed	closed	closed	
Drying Process After HPR										
	when				after HPR	after HPR	after HPR or after assembly	after HPR or after assembly	after HPR or after assembly	after HPR
	method				Class 10 area	Class 10 area	evacuation	evacuation	evacuation	Hang under cla HEPA
Assembly										
	Clean room class				10	10		10	10	10
Evacuation										
	Setup							for VT	for VT	
Speciality										
	Total time for the treatment	hrs				4	1 (Single) 6 (9-cell)	<--	1 (Single) 6 (9-cell)	
	Number of treatments needed for vertical test				1+6	2	1	1	1	
	Limitation on performance									

	More investigation needed on						Too much HPR degrades performance?		🟩	
									🟩	
	Ranking??								🟩	
									🟩	
	comments								🟩	
									🟩	
*	Sometimes, drying is performed in clean room (class 10) with beam pipe open during night.				allways				🟩	

Pure Water Facility													
		units	12/5/05 Decision	DESY	DESY 1cell	Jlab	KEK AR-east PW	KEK AR-east UPW	KEK (Nomura)	KEK (STF)	FNAL SMTF	ANL	Cornell
Pure water													
	City water prefilter size	um		none	10	5	None		Non	None			
	Carbon filter volume	m3				0,17	0,002		Non	0,05			
	Reverse Osmosis type	single /dual stages		single	single	single	single		Non	None	single		
	Permeate rate	lpm		12	7	22,7	20						
	Concentrate rate	lpm		6	3	9,1							
	Filter size	micron					0,1			0,1			
	Storage volume	liters		4000	500	3785,4	1300			450	3800		
									2				
								Use KEK AR-east PW					
Ultra-pure water													
	Circulation rate	lpm		20		127,3		-		10	19		
	Loop Resistivity	M-ohm-cm		18,2	18	18,2		18	18	18	18,2		
	Water Temperature	C		15,2	20	29		RT	RT	RT			
	Number of loops	#		2	1	2		2	1	3	1		
	Number of drops	#		5	4	8		3	5	4	7		
	ultrafiltration size	um		0,02	none	0,02		0,1	UF module	0,1	0,05		

	# post filters			1	1	2		1		1	2		
	post filter	um		0.2	0.2	0,54		0,1		0,1	0,2		
	UV frequencies			?	?	(194, 256)		-	UV	UV	1		
	Ion bed workers	m3			46L	0,17							
	Ion bed polishers	m3			46L	0,17							
	loop pressure	Atm		3	3bar	1,7				3	2,1		
	Production capacity	lpm		20				1	> 10	10			
	particle count				0.5 (0.2um)			30/cc (0.15µm)		None			
POU Chemistry													
	TOC	ppb		0,4-1,4	7-10	NA			~20	~10	NA		
	Bacteria count max	cfu		<=5		<50			50	5	>50		
	Bacteria count typical	cfu		<= 3		<50			<1	< 3	>50		
	Final Filter size	um		0,2		NA			Non	0,22	NA		
	Liquid particle count avg	counts/ml		0,08		0,09			NA	NA	NA		
POU HPR													
	TOC	ppb		<=1.41		<6			~20	10	<20		
	Bacteria count max	cfu		0		>500			50/cc	5	100/liter		
	Bacteria count typical	cfu		0		<50			<1/ml	< 3	10/liter		
	Final Filter size	um		0,04	00:04	0,04			0,2	0,22	0,04		
	Liquid particle count avg (0.3)	counts/ml		0,02		0,88			NA	NA	NA		
POU Part cleaning													
	TOC	ppb		<=1,46		NA		100 ~ 300		~10	NA		

	Bacteria count max	cfu		0		NA		500		5	NA		
	Bacteria count typical	cfu		0		NA		40/ml		< 3	NA		
	Final Filter size			0,02		NA		0,1		0,22	NA		
	Liquid particle count avg	counts/ml		1,04		NA		NA		NA	NA		

Heat treatment

		units	12/5/05 Decision	tol	DESY	Jlab	KEK New	FNAL SMTF	ANL	Cornell
Niobium Purification										
	Baseline Pressure (Room Temp)	mbar				<4e-7		NA		
	Ramp Rate	C/min				5		NA		
	Soak Temperature	C			1350	1250		NA		
	Pressure at Temp	mbar				4,00E-06		NA		
	Soak Time	min				180		NA		
	Ramp Down Rate	C/min				NA		NA		
	Pump Capacity	l/s (air)				3000		NA		
	Pump Capacity	l/s (hydrogen)				5000		NA		
	Chamber Hot Zone size	cm-length				160		NA		
		cm-width				58		NA		
		cm-height				58		NA		
	Experience	processes completed				40		NA		
High Temperature Baking/Annealing										
	Pressure Empty	mbar			<1e-8	<4e-7	1e-6 Torr	1,00E-07	5,00E-08	
	Ramp Rate	C/min				5	20	<5	1,7	
	Soak Temperature	C	600 ~ 800		800	600	750	750	600	
	Pressure at Temp	mbar	1,00E-06			5,30E-05	6e-4 Pa	<8.0E-08	5,00E-04	
	Soak Time	hrs	3			10	3	3	10	
	Ramp Down Rate	C/min			NA	NA	~8 hy *	<1	~3	
	Pump Capacity	l/s (air)				3000	10000	500		

	Pump Capacity	l/s (hydrogen)				5000		750		
	Chamber Hot Zone	cm-length				160	300	76	140	
		cm-width				58	ϕ 50	16	75	
		cm-height				58		16	75	
	Experience	processes completed				200	10	6	2	
	temp before opening	C	50				100			
	surroundings				Bare??	Bare??	Ti box			
Low Temperature (120C) Baking										
	Purpose				Q improvement		oxydized layer treatment	NA		
	Temperature	C	120	(--0,+15)	120-135	120	120	NA	120	
	Period	hr	10-48		48	48	48	NA	48	
	inside cavity				vacuum	vacuum	vacuum		vacuum	
	outside cavity				Nitogen	Atm nitrogen	air	NA	helium	
	Pump				oil free TMP		IP / TMP	NA	TMP (maglev)	

Evacuation system for various stages									
		units	12/5/05 Decision	DESY	Jlab	KEK AR-east	KEK STF	FNAL SMTF	ANL
UHV Systems for Cavity Evacuation									
	UHV pump type			TMP	TMP	IP	TMP	TMP	TMP (maglev)
	Pumping Speed (Air)	lps			60	250	250	100	300
	Roughing pump type			oil free	scroll	TMP + Rotary *	Scroll	Rotary Vane	scroll
	Roughing pump speed (Air)	lpm			250	250	100	100	600
	Typical Baseline Pressure	mbar			1,00E-08	3,00E-08	1,0E-07	1,00E-08	1,00E-08
	RGA	AMU			100	He detector	He detector	up to 100	He leak check
UHV Systems for Vertical Test							NA		
	UHV pump type			oil free TMP	TMP	No pumping		TMP	
	Pumping Speed (Air)	lps			60	-		100	
	Roughing pump type				scroll	-		Rotary vane	
	Roughing pump speed (Air)	lpm			250	-		100	
	Typical Baseline Pressure	mbar			1,00E-08	-		5,00E-09	
	RGA	AMU			100	-		up to 100	
	Evacuation method during test				Metal valve or active pump	Closed with metal valve		IP	
	Leak check after VT					He detector			
UHV Systems for Horizontal Assembly									
	UHV pump type			oil free TMP	TMP and ion pump		(IP + TMP) X 2	NA	TMP (maglev)
	Pumping Speed (Air)	lps			60/11		(250 / 250) x 2	NA	300

	Roughing pump type				scroll		Scroll X 2	NA	scroll
	Roughing pump speed (Air)	lpm			250		100 x 2	NA	600
	Typical Baseline Pressure	mbar			1,00E-08		1,0E-07	NA	1,00E-08
	RGA	AMU			100		He	NA	He leak check
	as of assembly							NA	

	Cleanroom for various activities											
		units	12/5/05 Decision	DESY	Jlab	KEK AR-east	KEK Nomura	KEK STF	FNAL SMTF	ANL	Cornell	
Cleanroom areas												
	Chemical area											
	Sub component chemistry area	m2		101.6	20,9	-		0,5	10	18		
	Cleanroom class	FED 209E		10 000	NR			1000	NR	NR		Fed 209E
												1
	Subcomponent degreasing area	m2		2.0	62,8	-			8	20		10
	Cleanroom class	FED 209E		10 000	NR				1.000	1000		100
												1000
	Cavity chemistry (BCP) area	m2		0.8	22,6				17	18		10.000
	Cleanroom class	FED 209E		10 000	100				NR	NR		100.000
	Cavity Chemistry (EP) area	m2		35.4	36,5		30	120	17	18		
	Cleanroom class	FED 209E		outside cleanroom	10.000		Non	NR	NR	NR		NR-not rated
	Cavity HPR area	m2		1,5	22,6	9	12	3	9	23		
	Cleanroom class	FED 209E		10	100	1000	1000	100	100	100		
	Cavity assembly area for vertical test	m2		23	6,4	10		14	18	15		
	Cleanroom class	FED 209E		10	10	10		10	10	100		

	String assembly area	m2		103,7	45,1			14	67	20		
	Cleanroom class	FED 209E		10 / 100	100			ISO 4	10	100		
	Vertical stand area	m2			88,8	12		NA	10	?		
	Cleanroom class	FED 209E			100.000	NR			NR			
	Module assembly area	m2			117,8				100	linac tunnel		
	Cleanroom class	FED 209E		none	NR			NR	NR	NR		
	Module staging area	m2			120,8				110	linac tunnel		
	Cleanroom class	FED 209E			NR			NR	NR	NR		
L340xc												

Component Cleaning Requirements

		12/5/05 Decision	DESY	Jlab	KEK AR-east	KEK AR-east	KEK STF	ANL
Cavity Qualification								
	Input coupler, field probe assemblies		Untrasonic + UPW rinse	Ultrasonic + UPW rinse+ nitrogen dry	CP + Ultrasonic in PW	CP + HPR(PW) + Ultrasonic in UPW	CP + HPR(PW) + Ultrasonic in UPW	Ultrasonic+manual HPR
	UHV components		ultrsaonic +UPW + annealing	Ultrasonic + UPW rinse+ nitrogen dry		Ultrasonic in UPW	Ultrasonic in UPW	same
	Nut/Bolt Hardware		dish washer + Ultrasonic +UOW	Ultrasonic + UPW rinse+ nitrogen dry		Non	None	same
String Assembly								
	input coupler, pickup		UPW Ultrasonic baking	Ultrasonic + UPW rinse+ nitrogen dry	CP + Ultrasonic in PW			Ultrasonic+manual HPR
	UHV components		ultrsaonic +UPW + annealing	Ultrasonic + UPW rinse+ nitrogen dry				same

	Nut/Bolt Hardware		dish washer + Ultrasonic +UOW	Ultrasonic + UPW rinse+ nitrogen dry				same
Horizontal Test (Chechia)								
	UHV components							
	Nut/Bolt Hardware							
Module Fabrication								
	UHV components			Ultrasonic + UPW rinse+ nitrogen dry				
	Nut/Bolt Hardware			Ultrasonic + UPW rinse+ nitrogen dry				

TTC-Meeting at KEK 2006

Sept.25-28, 2006

Analysis technique of the HF-H2SO4-electrolyte

Christian Hartmann
Henkel Lohnpoliertechnik GmbH

Location	**Austria** **Waidhofen/Thaya**	**Germany** **Neustadt-Glewe**	**Hungary** **Györ**
Foundation	1977	1996	1994
Employees	40	40	7
Business Activities	Stainless steel technology, pickling, passivation, electropolishing, clean room techniques	Stainless steel technology, pickling, passivation, electropolishing, clean room techniques	Stainless steel technology, pickling, passivation, electropolishing
Production Area	approx. 6.500 m2	approx. 4.500 m2	approx. 2.000 m2
Clean Rooms	Clean rooms 100.000, 10.000, 100 and 10 (US-Federal Standard 209 D)	Clean rooms 100.000, 10.000, 100 and 10 (US-Federal Standard 209 D)	
Electropolishing Baths	up to 80.000 ltr.	up to 23.000 ltr	up to 9.000 ltr.
Pickling Baths	up to 60.000 ltr.	up to 23.000 ltr	up to 9.000 ltr.
Electropolishing Machines	for internal ep of tubes with diameters 1... 350 mm and lengths up to 18.000 mm	for internal ep of tubes with diameters 1... 350 mm and lengths up to 18.000 mm	
Auxiliary Supplies	Deionic ultra pure water installation (18MWcm at 25°C, TOC < 50 ppb, KBE< 10/100 ml, particle filter 0,1 µm), cryo source for ultra pure nitrogen (quality N2 5.0, particle filter 0,003µm)	Deionic ultra pure water installation (18MWcm at 25°C, TOC < 50 ppb, KBE< 10/100 ml, particle filter 0,1 µm), cryo source for ultra pure nitrogen (quality N2 5.0, particle filter 0,003µm)	Reverse Osmose Installation (4MWcm at 25°C)

Analysis technique

- **Titration**
- **ICP-OES** (inductively coupled plasma- optical emission spectroscopy)
- **IC** (ionic-chromatography)
- **TOC** (total organic carbon)
- **NMR** (nuclear magnetic resonance)
- **FTIR-ATR-probe** (Fourier transformation infrared spectroscopy –attenuated total reflection)

Production process of Henkel electrolyte

- **Warm test**

  - mixing HF-acid (75%) with *normal* water

  => HF acid (46%)

  - mixing HF-acid (46%) with H2SO4 (98%) *without cooling* in the rate of 1/9

  - the reaction ist hot – *more than 60°C and steaming*

  - Rust impurities are possible

- **Cold test**

  - mixing HF-acid (75%) with *deionised* water

  => HF acid (46%)

  - mixing HF-acid (46%) with H2SO4 (98%) *cooling under 20°C* in the rate of 1/9

  - less steam and storing always under 20°C

Titration (volumeric analysis)

Total Acidity per titration (measured by Solvay-Lab):

Warm test	Cold test	
84,8 % (84,64 - 85,1)	84,4% (84,0 - 84.6)	H_2SO_4

Total acid as H_2SO_4, fluoride bounded to calcium - 6 titrations

\+ automated
definition of total ionic concentration possible

\- you must know what you want to analyse
(selection of titration solvent)

Costs: approx. 10.000 - 15.000,- €
Producer: Metrohm, Schott, Mettler-Toledo

ICP-OES (optical emission spectroscopy)

Principle: the testsubstance is ionised in an argon plasma (6000K–12000K), the effect is an optical emission you can analyse in a spectrometer

\+ simultaneous multielement analyse upto 70 elements (qualitative und quantitative)
better repeatability than AAS because insensitivity of interferences

\- no information about molecular characteristics

costs: approx. 60.000,-€
producer: PerkinElmer, Varian, Spectro, Thermo…

ICP-OES (optical emission spectroscopy)

by Solvay (Thermo ICAP 6500)

results in mg/kg

	warm	cold
Nb	<0,1	<0,1
Ta	<0,1	<0,1
Na	1,4	1,4
K	1,1	1,5
Mg	0,4	0,2
Ca	0,9	1,2
Sr	<0,1	<0,1
Ba	<0,1	<0,1
Ti	<0,1	0,3
Zr	<0,1	<0,1
Fe	**32**	**14**
Cr	0,15	0,19
Ni	0,14	0,14
Mn	0,19	0,15
Cu	<0,1	<0,1

	warm	cold
Zn	<0,1	<0,1
Pb	<0,1	<0,1
Cd	<0,01	<0,01
V	<0,1	<0,1
Mo	<0,1	<0,1
W	<0,1	<0,1
Sn	<0,1	<0,1

ICP-OES (Varian with Varian 735-ES)

Probenkennung	**Fe 238.204**	**K 766.491**	**Mg 285.213**	**Mn 257.610**	**Mo 202.032**	**Na 588.995**
	ppm	ppm	ppm	ppm	ppm	ppm
Warm 1	46.02	0.3915	0.4947	0.2493	0.0515	1.243
Warm 4	49.41	0.3790	0.5541	0.2720	0.0563	1.397
Cold 1	22.38	0.3845	0.2267	0.1375	0.0244	1.138
Cold 2	21.21	0.2513	0.2541	0.1353	0.0389	1.232
Cold 4	18.94	0.3104	0.2163	0.1269	0.0171	1.127
Cold 5	19.12	0.4483	0.2231	0.1309	0.0141	1.129
Cold 6	18.40	0.5197	0.2179	0.1256	0.0214	1.123

IC- Ionic chromatography

Principle: analysis technique for ionic species (anions and cations). Based on the characteristic of ions bounded to polar fixed adsorbent materials (ion exchange resin). The ions are different strong bounded, kind and size is critical for the species. Stronger bounded ions appear later in the chromatogram. The ion detection is mostly the result from the conductivity measurement.

\+ Simultaneous multi anions analysis + concentration definition
Good reproducible
More from SACLAY: F-, sulfat, SO_3F^-

\- Only ion detection - no information about molecules
Only diluted solutions

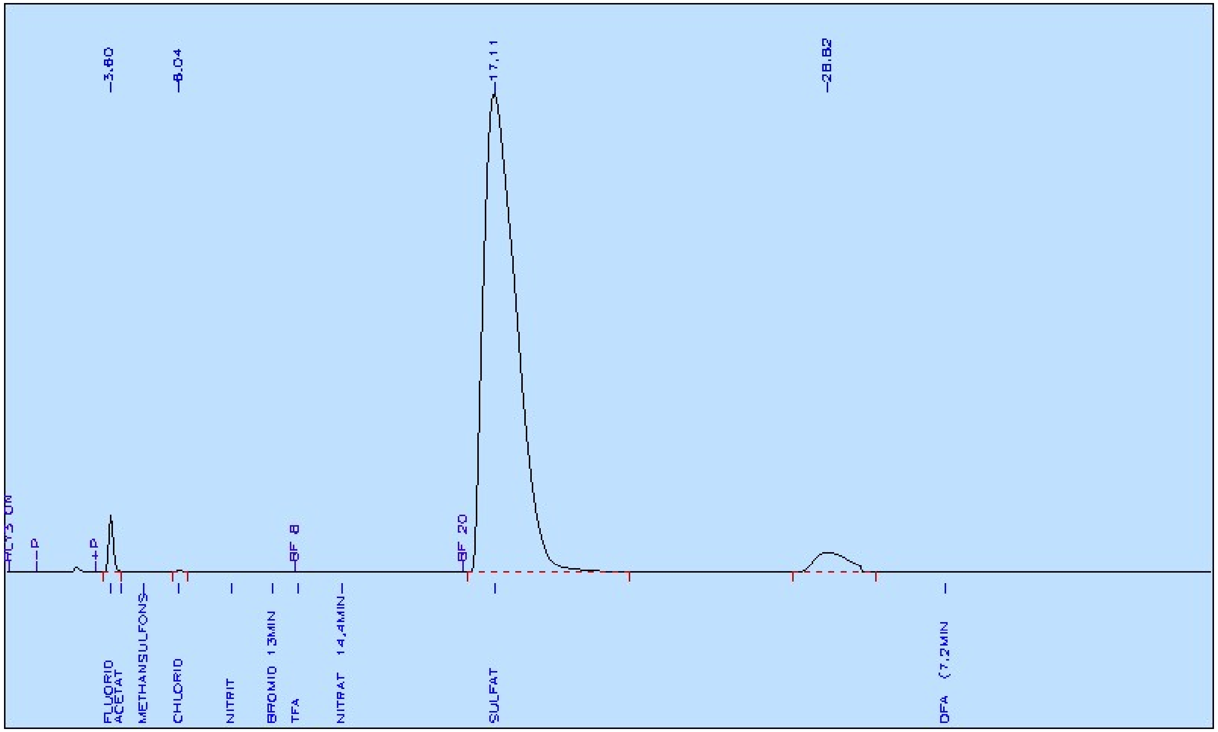
Costs: >15.000,-€
Producer: Thermo, Dionex, Shimadzu, Metrohm...

IC- Ionic chromatography

Solvay Lab:
Dionex IC500

warm cold
0,73% 0,77% F-
2,76% 3,05% FSO3H

total fluoride is incapabel of measurement by IC – no total hydrolysis of FSO3H in H2SO4 and HF



IC- Ionic chromatography

Solvay Lab:

Measurement of total fluorid is possible by destillation

=> complete disintegration

Metrohm Ionicmeter Typ 692 (fluorid. standard + TISAB)

Result:	**Warm**	**Cold**	
	2,62%	2,99%	total F-
=>	2,75%	3,14%	HF

Infraserv Knappsack Lab:

Warm1		**Cold5**
82%	81%	H2SO4
1,0%	1,1%	F-
3,9%	4,0%	FSO3H

TOC (total organic carbon)

Question: Are there any organic impurities in the electrolyte?

Solvay Lab

TOC-Laboratory Analyser: Shimadzu

Calibration: 5mg/l C

Result:	**Warm**	**Cold**
	30 mg/kg	59 mg/kg

the result is near the limit of quantitation!!

NMR (nuclear magnetic resonance)

Principle: Atoms with magnetic dipole arrange themselves in an external magnetic field (parallel or anti-parallel). These conditions differ by a certain amount of energy. The transition can be enforced by irridation of electromagnetic radiation at a specific frequeny: Lamorfrequency. The necessary frequency for the transition is measured. The result delivers conclusion about the electronic surrounding of the examined atomic nucleus and thus about the structure of the molecule.

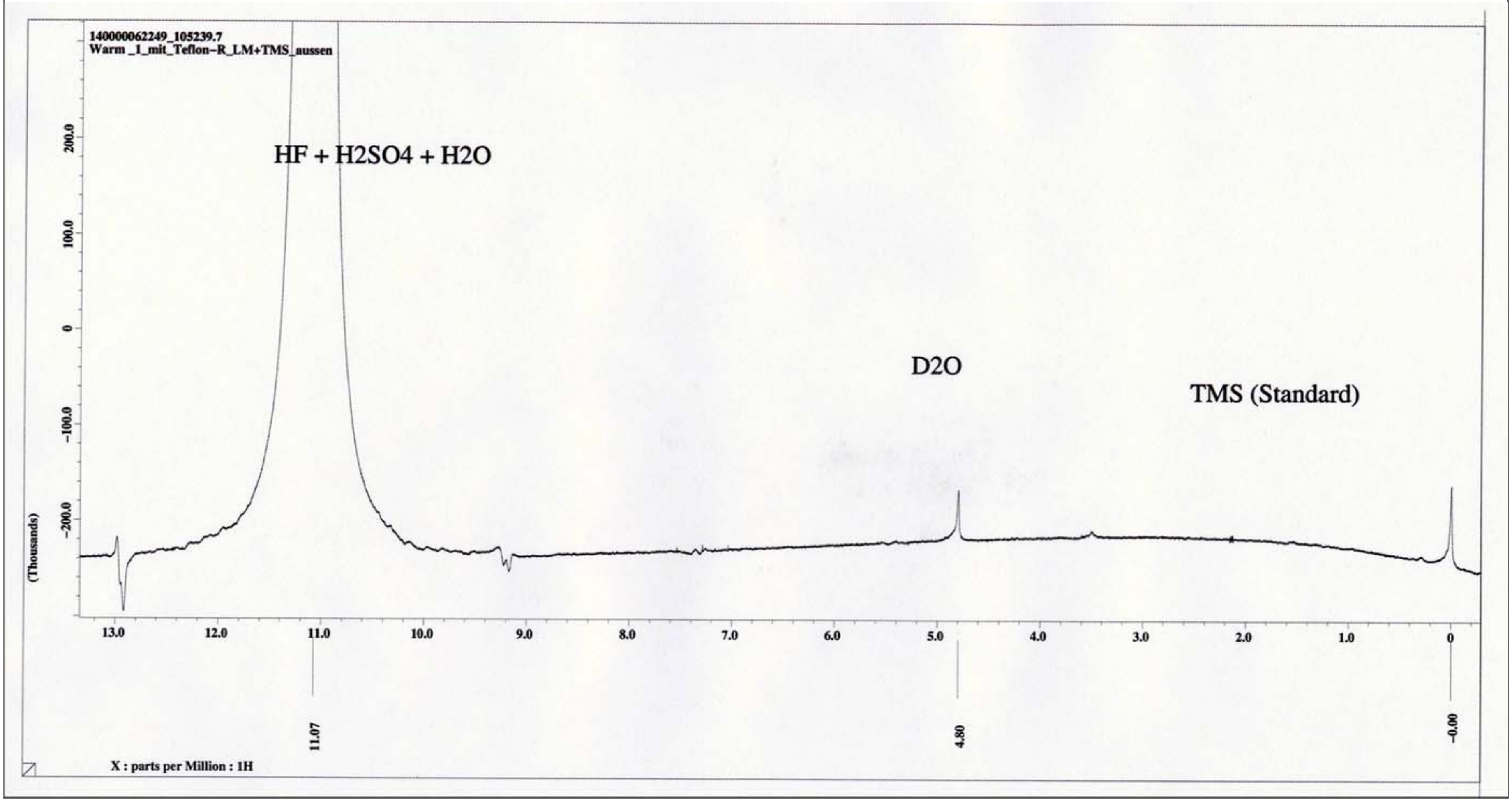
\+ Molecular structures are precisely determinable
Interaction between molecules can be analysed

\- Analysed atomic grade requires magnetic dipol moment (at 1H, 19F)
Gases are not analysable

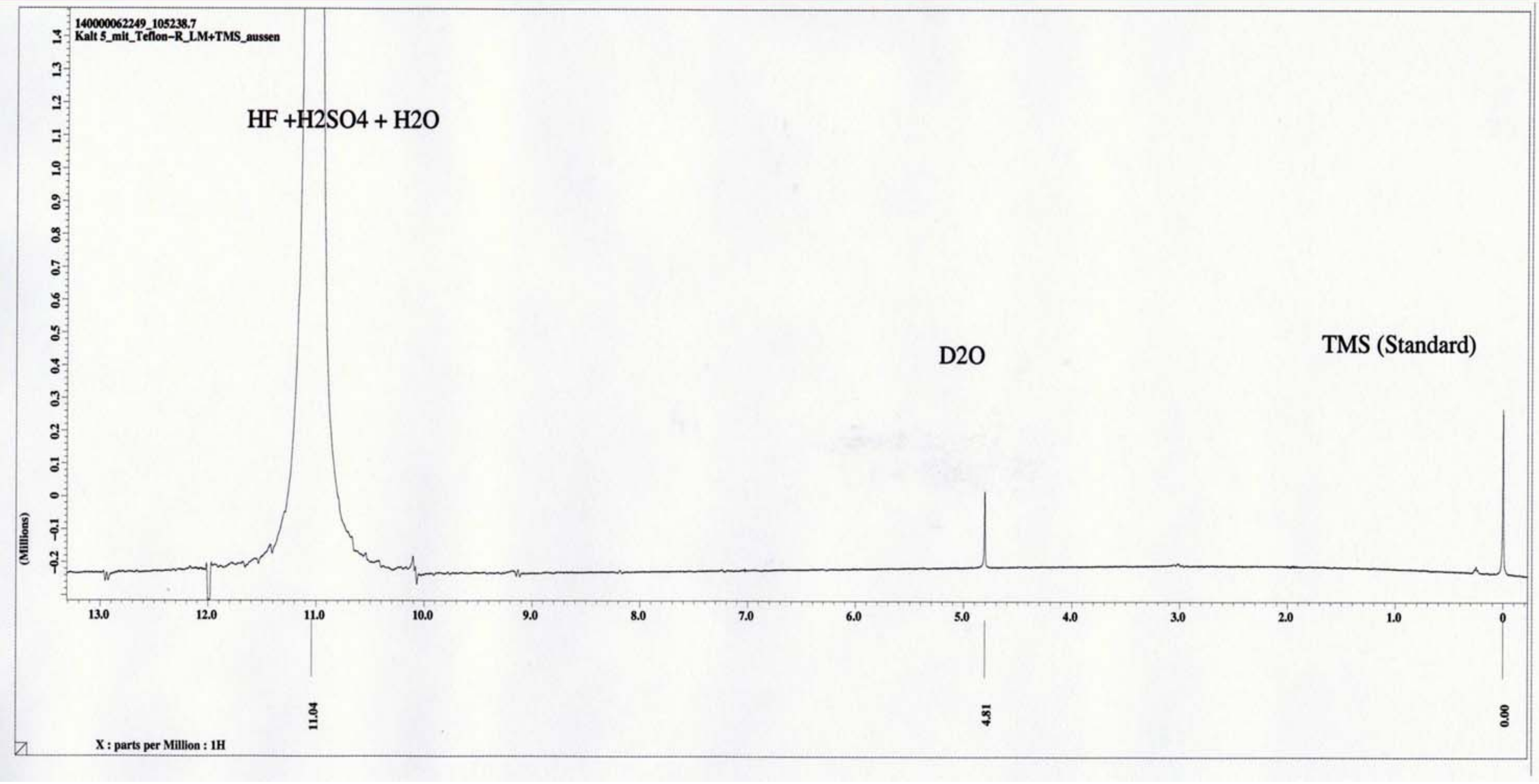
Costs: >>100.000,-€ to some extent up to 4Mill. €; external: each measurement 170,-€ - 600,-€ according to measuring time

Producer: Brukeroptics, Varian,...

NMR Infraserv Warm (1H-NMR)



NMR Infraserv Cold (1H-NMR)



(FT)IR (Fourier transformation infrared) Spectroscopy

Principle: Wave lenghts dependent stimulation of bonds between atoms for mechanical oscillation through absorption of light within the IR-range (wave lenghts of approx. 0,8-500 µm). The hereby obtained interferogrammes contain the complete radiation absorption of the trial after wave length and intensity as Fourier sum of all spectral lines. Resultant is a better dissolving ability as with the classic IR-spectroscopy.

\+ Direct measurement of properties of the electrolyte
Qualitative and quantitative analyses are possible

\- Main problem : electrolyte resistant glas materials for cuvettes must be found (and all the same transparent in the IR-range)

Costs: >15.000,-€
Producer: Varian, Brukeroptics, Shimadzu, Thermo,...

ATR-Probe (attenuated total reflection)

Principle: Total reflectance at the interfaces is used. At total reflectance the radiation intrudes a little into the surrounding medium. Depending on whether or not the trial absorbes radiation the intensity of the reflected radiancy is therewith influenced. The result is a transmission spectrum similar to the reflection spectrum.

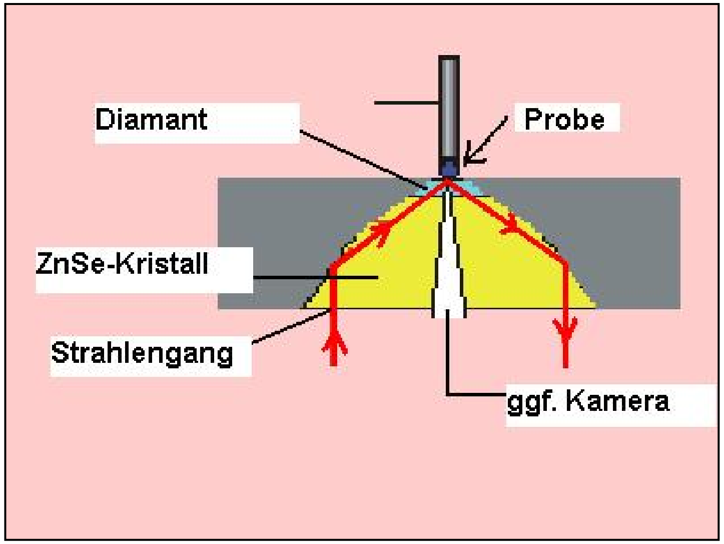
\+ Direct measurement of properties of the electrolyte
Diamond resistant against aggressive media,
Mobil

\- Contact between electrolyte and diamond mounting must be prevented (Teflonseals ?)

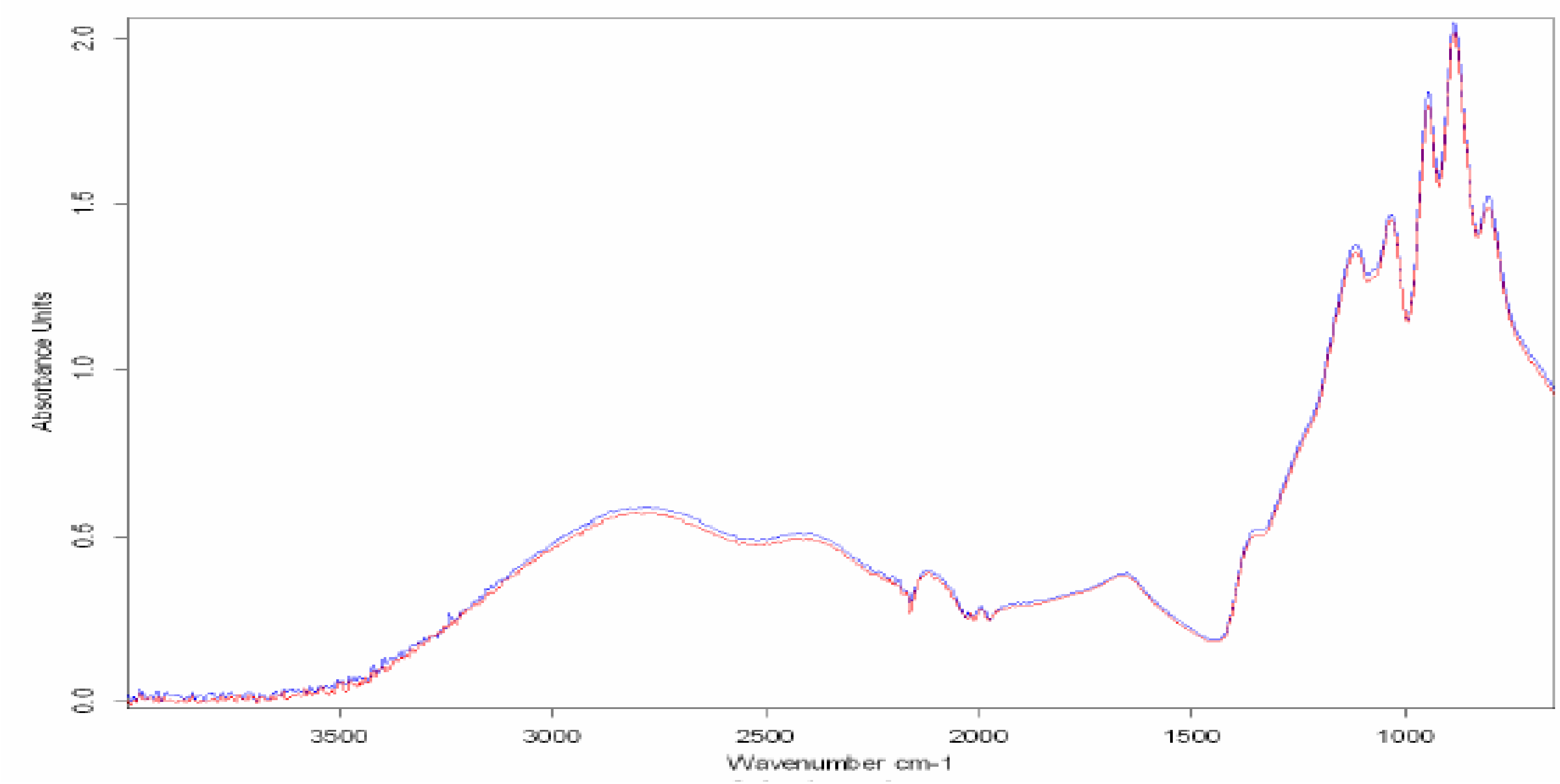
Costs: only ATR-unit : 10.000-15.000,-€
Provider: HiTec Zang, C3

ATR-Probe (attenuated total reflection)

HiTec Zang ATR-Probe used by RWTH Aachen



ATR-Probe (RWTH Aachen with HiTec Zang)



Summary

• Titration	+ total acidity
• ICP-OES	+ all elements (Nb, Fe,...)
• IC + destillation	+ Ions (SO4, F-, FSO3H)
• TOC	+ organic impurities
• NMR	+ organic impurities ???
• FTIR-ATR-probe	???

To Do: analyse more tests!!! (with impurities and Nb)

Compare the tests with the results of the cavities