MINUTES

OF THE "PRECOS" PROJECT MEETING

St.Petersburg 2 June 2010

**MEETING PARTICIPANTS**

**Collaborators:**

Marc Barrachin - IRSN

David Bottomley - EC, ITU

Sieghard Hellmann - AREVA NP GmbH

Manfred Fisher - AREVA NP GmbH

Pascal Piluso - CEA (instead of Fransoise Defoort)

Invited persons:

Gert Langrock - AREVA NP GmbH

**Operation Agent:**

**NITI: SPbTU:**

Khabensky V.B. Gusarov V.V.

Bechta S.V.

Granovsky V.S. **SPb SETU “LETI”**:

Krushinov E.V. Vavilov A.V.

Vitol S.A. Lopukh D.B.

Kotova S.Yu. Almjashev V.I.

Kalyago E.K. Martynov A.P.

Sulatsky A.A. Kirillova S.A.

Lysenko A.V.

Poldyaeva I.Yu. **SPbSU:**

Shuvalov S.V. Kalinin B.D.

**IVTAN:** **SPbPTI:**

Sheindlin M.A. Tugova E.A.

**IMC RAS:**  **NATI Research:**

Almjasheva O.V. Knauf V.V.

Bugrov A.N.

**Objective of the meeting:**

The main objective was the presentation of reports on the work performed within the framework of PRECOS project and discussion of the future work plans.

**Agenda:**

1. Welcome and organizational issues S.V. Bechta
2. PRECOS project status S.V. Bechta
3. PRECOS project activity in the 5- 8 quarters V.V. Gusarov
4. Progress report on UO2-SiO2 system V.I. Almjashev
5. Progress report on UO2-CaO system E.V. Krushinov
6. Results obtained with laser pulse heating facility M.A. Sheindlin
7. Progress report on ZrO2-FeOy system study O.V. Almjasheva
8. First results on UO2-FeO-CaO, UO2-FeO-SiO2 system S.U. Kotova
9. Problems and perspectives of SEM/EDX and chemical

analyses of corium samples S.A. Vitol et al.

1. Round-Robin SEM-EDS analyses of a corium sample

from the Vulcano UB-V6 experiment P. Piluso

1. Status of CORPHAD & PRECOS publications V.I. Almjashev
2. Plans for 9-12 quarters V.V. Gusarov
3. Discussion All
4. Next meeting All
5. Concluding word S.V. Bechta

**1. Welcoming address, agenda, organizational issues – S.V. Bechta.**

**S.V. Bechta** opened the meeting, greeted participants and proposed them to get acquainted with the Agenda and sign Minutes of the previous meeting. M. Sheindlin requested to schedule his presentation for earlier in the day instead of that by O. Almjasheva.

**2. PRECOS project status - S.V. Bechta.**

In his presentation **S.V. Bechta** told about organizations and participants involved in the project, its aims and expected results. Also, he reported about the corrections made to the project and about the increase of the amount of research performed by the group of M. Sheindlin at IVTAN. S. Bechta refreshed the Experimental Matrix, stressed the delay in performing the work as per Items 1 and 2 of the Matrix and named the reasons. Then the working plans for the 3rd year were discussed, and the status of 4 actions adopted by the previous meeting reported.

1. concerning heating pureSiO2 to 2200°С and measuring its stoichiometry. SiO2 evaporation has been investigated. The decrease of samples’ mass at different temperatures has been determined. The stoichiometry measurement procedure has not been worked out and is not totally clear. It has been proposed to terminate this action.

2. Concerningpreparation of standard (reference) samples for the SEM/EDX calibration. The question is about the influence of samples preparation quality on the elemental analysis of samples from the tests with the UO2-CaO system and about determining the influence of the changed beam power on Ca fluorescence. A bank of reference samples has been prepared. The influence of the changed electron beam power has not been investigated due to the problems with the electron microscope adjustment. The work will be continued.

3. Concerningthe necessity of comparing PRECOS results with the data published by Prof. Yu.B. Petrov on miscibility gap (MG) in theZrO2 – FeOy system. Experiments with melt compositions in the supposed MG have not been performed. The work will be continued.

4. Concerning the help from M. Barrachin with thermodynamic calculations for a paper entitled *Phase equilibria in the FeO1+x–UO2-ZrO2 system*. The paper has been published. M. Barrachin was thanked for his help with preparing the paper. Action completed.

**S.V. Bechta** introduced a new collaborator from CEA. Instead of Françoise Defoort, CEA will be represented by P. Piluso.

**P. Piluso** made a remark that F. Defoort has changed the field of research and at present is involved in biomass studies.

**D. Bottomley** thanked M. Barrachin for his help with paper preparation.

**3.PRECOS project activity in the 5-8 quarters - V.V. Gusarov**

**V.V. Gusarov** told about the work plans for Quarters 5-8. He said that his presentation should be regarded as an introduction to the presentations to follow. Speaking about the ZrO2-FeO diagram a special attention should be paid to the fact that the results and the shape of the diagram are being refined.

*No questions followed.*

**4. Progress report on UO2-SiO2 system - V. Almjashev**

**V. Almjashev:** Proposed to revere the memory of M.D. Tolkachev who had been responsible for the SEM/EDX analysis in the project and passed away in 2009. After the minute of silence, V.Almjashev proceeded to the technical part of his report and presented experimental results of the investigations. The performed work has yielded the refined liquidus and tie lines in MG. According to the Matrix, 7 points had to be investigated, while the actual number has already reached 40. After generalizing all the obtained data, it will be possible to start preparing a publication.

*Questions:*

**D. Bottomley:**

The work has been done very thoroughly. What is the size of your specimens in comparison with those used by Lungu?

**V. Almjashev:** Lungu used 10-mm long boat-shaped vessels of iridium or tungsten. Basically, the sizes of his and our specimens are comparable. Lungu was determining the composition of liquids at the monotectic temperature, however he did not determine the bimodal boundaries.

**D. Lopukh:** What does the red line on the diagram designate?

**V. Almjashev:** It designates the Lungu’s data.

**D. Lopukh:** Have you made any comparison with the Yu.B.Petrov’s data?

**E. Krushinov:** You may remember that he has not done any points, so there are no quantitative data available for comparison.

**S. Hellmann:** Did you experience any problems related to evaporation?

**V. Almjashev:** We covered the crucibles with covers. The compositions at the top and bottom of crucibles are identical.

**S. Hellmann:** Where did condensing take place?

**V. Almjashev:** Condensing occurred near the covers. Two effects are involved: on the one hand, evaporation/condensation, on the other hand, good moistening of crucible walls with the melt.

**S. Bechta:** Was there a shift in the composition compared to that of the charge?

**V. Almjashev:** There was a shift during the charge preparation, since there was a too big difference in atomic weights. The total shift was not big, just around 5%.

**S. Bechta:** Was it U-biased?

**V. Almjashev:** The evaporation of U and Si at this temperature is approximately the same, therefore the shifts are possible both ways. We attempted to evaluate the bulk composition using polished sections instead of the charge, but it was very difficult due to the components distribution inhomogeneity.

**D. Bottomley:** Slide 12. MG is asymmetrical and the liquidus line is almost straight, maybe it should be curved?

**V. Almjashev:** This series of points is trustworthy; if there is a curve, it is very insignificant.

**D. Bottomley:** In the case of a shift towards SiO2, the system tends to shift towards a higher viscosity level. This is the reason of MG asymmetry, and usually the steep slope is observed on the SiO2 side, while you have quite the opposite. Maybe, it is not the real picture, but an experimental shift. Maybe, it happens because of the analytical error, for instance, problems with correct selection of areas for analysis? It might be interesting to have longer exposures and calculate the trend of the liquidus and binodal curves.

**V. Almjashev:** Probably, equilibrium has not been reached here and longer exposures are required. Indeed, the majority of experimentally determined binodals are SiO2-biased. Traditionally, some effects occurring in the stratifying systems are disregarded (the composition is determined for the areas that do not correspond to the composition of the liquid on the SiO2 side) and MG on the SiO2 side flattens in the opposite direction. We are planning to make a thermodynamic calculation of the curves’ trend soon.

**D. Lopukh:** What are the limits of the error for the points plotted on the diagram?

**V. Almjashev:** 30°C in terms of temperature, and 2 rel. % in terms of composition.

**P. Piluso:** Have the MG data been obtained for the stratified layers? Why VPA IMCC hasn’t been used for the binodal temperature determination?

**V. Almjashev:** Yes, the data have been obtained for the stratified layers. VPA IMCC cannot be used in this case because in view of the big size of the pool, large mass of the melt, its viscosity and inhomogeneity, it is difficult to reach a quenching rate that will yield conditions close to the equilibrium.

**E. Krushinov:** We have conducted such a test, PRS8, a video record of which showed the emergence of the second liquid at the melt surface, but it was impossible to link temperatures and compositions in the course of these processes. When crystallization of the darker liquid started, the temperature definitely coincided with the monotectic temperature.

**M. Barrachin:** Are you sure that in the MG tests it was stratification, but not gravitational stratification of the uranium-rich phase?

**V. Almjashev:** We realize the possibility of gravitational effects, therefore we increased the annealing time and achieved high cooling rates in order to minimize said effect.

**M. Barrachin:** When did you take samples when measuring Tliq?

**E. Krushinov:** Immediately before the measurement.

**S. Bechta:** You said that it was necessary to work on a paper, but when the report will be ready?

**V. Almjashev:** Technical materials are all ready.

**S. Bechta:** One month will be required for preparing the Russian version of the report and another month for preparing the English version.

**5.Progress report on UO2-CaO system - E.V. Krushinov**

The author presented results of the preformed experimental investigations. They made it possible to refine the liquidus line and composition of the UO2–based limiting solid solution.

*Questions:*

**D. Lopukh:** At what speed the ingot was drawn out of the inductor?

**E. Krushinov:** 9 mm/h.

**M. Barrachin:** In which acids the quenched melt samples were dissolved?

**S. Vitol:** We managed to compose a mixture of acids in which experimental samples dissolved completely.

Method 1: samples were dissolved in a mixture of sulfuric and phosphoric acids;

Method 2: at first, the samples were fused with a specially selected flux and then transferred into a solution.

**M. Barrachin:** At which temperatures the compound CaUO3 is stable?

**E. Krushinov:** This compound forms in the eutectic nucleus, we observe it, it does not decompose, maybe, it is stable all the way up to Tsol.

**M. Barrachin:** Your data should be compared with those of Pialoux, who studied this diagram in the low temperature domain. Using an X-ray high temperature attachment, he found this compound to decompose at 1050°С. He never observed Ca2UO4.

**S. Bechta** (addressing M. Barrachin): How did he prepare his samples?

**M. Barrachin:** I do not remember; I should send you a copy of his work.

**Action (5) M. Barrachin will send a copy of the paper by Pialoux on the studies of the subsolidus domain of the UO2-CaO diagram.**

**E. Krushinov:** We performed the XRF analysis (X-ray fluorescence) almost on the eve of the presentation and observed this compound in the area of the last liquid crystallization. The melt cools down quickly, therefore the existence of Ca2UO4 around Tsol may be supposed. It should be noted that according to the experimental matrix, we do not investigate the subsolidus domain of the diagram.

**S. Bechta:** Our diffractometric measurements were made at the room temperature, while Pialoux’s measurements had been made at a high temperature, and his results deserve a very careful study.

**M. Barrachin:** First slides. At first, you have mentioned diagrams showing solubility of 47 and 30 mol.%. For NUCLEA, we’ve accepted 30%, and solubility was comparable with your results, i.e., there was no increase in solubility.

**S. Bechta:** According to our preliminary data, CaO solubility is 52.8 mol.%.

**E. Krushinov:** We share an opinion that the solid solutions area lies in a narrow temperature range and adjoins the liquidus curve.

**P. Piluso:** Shared his experience of samples preparation for SEM/EDX analysis. A big problem with preparing samples for analysis is determined by the active CaO/ambient atmosphere interaction (i.e., with moisture and CO2), which leads to the destruction of a sample (swelling, cracking). We tried applying oils, but since this technique is difficult to realize in the glove box, we decided to switch back to dry polishing. Also, gasoline can be used for the purpose, however, it is forbidden in France.

**P. Piluso:** How the bottom crust was measured in the tests?

**E. Krushinov:** With a tungsten probe.

**S. Hellmann:** Your CaO melting point is quite high in comparison with the results by other authors.

**E. Krushinov:** M. Sheindlin will tell about that in more detail, as he preformed these investigations.

**M. Barrachin:** Two types of measurements are employed, that is with and without crucibles. Your crucible-free measurements applying the LPH method coincide with those in the solar furnace. The measurements made in crucibles show much lower temperatures.

**6.Results obtained with laser pulse heating (LPH) facility - M. Sheindlin**

In his report, M. Sheindlin told about the preparation of a complex of experimental equipment and presented the first results of investigations of the Zr-O and ZrO2-FeO systems which made it possible to refine the CaO melting temperature. In conclusion, he noted the continuing problems with licensing the laboratory for handling natural uranium.

*Questions:*

**P. Piluso:** What was the quenching rate?

**M. Sheindlin:** We are talking about the cooling rate which may be evaluated from the diagram. We can change it arbitrarily; power can be reduced to some value different from zero, usually it amounts to 20% of the maximum power. We are not in favour of rapid cooling, on the contrary, we extend this process in order to avoid supercooling.

**M. Barrachin:** Can it be done under pressure and what this pressure can be?

**M. Sheindlin:** Yes, it is possible. We have a similar facility with a 3kBar pressure in the cell.

**M. Barrachin:** I propose to repeat several points from the UO2-SiO2 system applying the LPH method, but this time do it under pressure in order to prevent evaporation of SiO2. The data for the 50:50 mol.% area significantly differ from those yielded by the thermodynamic calculations using NUCLEA.

**M. Sheindlin:** If the opinion will be expressed by the colleagues that these data are of interest, we can study the compositions of interest within the UO2-SiO2 system.

**S. Bechta:** When will the license for uranium handling be obtained?

**M. Sheindlin:** According to the moderately optimistic expectations, it may happen by the end of the year. Also, we have to transport the samples from “Luch” Scientific-Production Association to our laboratory, and the license may be ready by the end of October.

**P. Piluso:** The data on ZrO2 solidification – are these predicted or experimental data?

**M. Sheindlin:** These are experimental data.

**D. Bottomley:** These results are interesting concerning the ZrO2-FeO system, if a transition is made to Fe3O4, Fe2O3 under oxygen pressure. Speaking about UO2-СаO, can you possibly measure the samples with low UO2 content?

**S. Bechta:** But these are different diagrams, we should complete the work with ZrO2-U first.

**D. Bottomley:** I have a proposal to have the CaO melting point measured, and then to measure another point after adding a small amount of uranium.

**M. Sheindlin:** We can make the measurements if the samples will be available.

Action (5) The Operation Agent will prepare and present proposals concerning samples preparation and measurement of additional compositions by the LPH method.

**7.Progress report on ZrO2-FeOy system study - O. Almjasheva**

The work performed with the ZrO2-FeOy system has been reported.

*Questions.*

**D. Lopukh:** What are the problems with equipment adjustment?

**O. Almjasheva:** The absence of sample holders made from precious metals (iridium).

**S. Bechta:** You’ve been talking about work planning; we learned about the start of the work, but when will it be completed?

**O. Almjasheva:** We hope to complete this work on time. By now, the eutectic and subsolidus domains of the diagram have been investigated. The main problem with performing measurements in the high-temperature domain is in the absence of precious metals.

**S. Bechta:** These investigations had to be completed already.

**O. Almjasheva:** I think, we’ll be able to complete the work by October.

**P. Piluso:** Can the sol-gel method be used for uranium samples preparation?

**O. Almjasheva:** I do not work with uranium samples.

**M. Barrachin:** You’ve talked about MG. Were these Yu.B.Petrov’s or your data?

**O. Almjasheva:** These were Yu.B.Petrov’s data and the question about MG still stays open.

**8. First results on UO2-FeO-CaO and UO2-FeO-SiO2 systems- S.U. Kotova**

The author presented experimental results of the performed research and outlined plans for the future tests.

*Questions.*

**D. Bottomley:** There’s a misprint in Slide 21 – there should be written “eutectic” composition instead of the “monotectic”. It is obvious that quite a volume of work has been done on the UO2-FeO-SiO2 system. However, the diagram shows that the loaded charge compositions are different from the presented tie-lines.

**S. Kotova**: Yes, a shift in samples’ composition is due to the interaction with the molybdenum crucible which caused melt depletion in Fe, and also due to a significant evaporation of silicon oxides (presumably, of the monoxide mainly).

**S. Bechta:** We have not placed the average composition point on the tie-line, we’re just showing the supposed direction of the shift from the initial charge.

**E. Krushinov:** Micrographs show SEM images, from which it follows that equilibrium was not achieved during annealing, but at the same time the regions indicating melt stratification were registered. That enabled us to plot tie-lines for both light and heavy liquids. It is practically impossible to plot bulk compositions on the diagram. The slide also shows the ternary eutectic composition registered in GPRS36 test.

*There followed a discussion between D. Bottomley, E. Krushinov and V. Almjashev concerning difficulties with determination of compositions of the light and heavy liquids due to inhomogeneity of the ingot.*

**M. Barrachin:** Melt contamination with the crucible material has been noted. How bad is it qualitatively?

**V. Almjashev** shows Slide 16 about GPRS33, where Fe+Mo are located at the crucible bottom in small quantity.

**E. Krushinov:** The purchased FeO reagent contains 30% of Fe2O3 and the transition of Fe+3 into Fe+2 requires Femet. It is this metallic iron that reacts with molybdenum. The crystallized oxidic melt shows the presence of molybdenum in trace quantities.

**V. Almjashev:** Fayalite should be synthesized for investigations.

**S. Bechta:** Do you mean that this will solve the problem of interaction with the crucible?

**V. Almjashev:** It can at least ease the problem.

**M. Barrachin:** Where does silicon oxide deposit in the furnace?

**A. Lysenko:** In the Galakhov microfurnace it deposits on cold surfaces when penetrates through the crucible cover.

**D. Bottomley:** Has the aerosol composition been determined during the VPA IMCC?

**S. Kotova**: Yes, each time we endeavour to completely analyze the fused products for composing the elemental material balance. In PRS13 the quantity of collected aerosols was big and their composition will be identified, in contrast to PRS14, in which the quantity was low, their collecting was impossible and all the deposits were washed away from the transport lines (i.e., only their mass was determined from the difference between masses of the dirty and clean transport lines). Collecting aerosols from the large area Petryanov filters will most likely fail, as their small quantity is spread across the large area of these filters.

**E. Krushinov:** The final report will contain the elemental composition of all samples, aerosols included, as much as possible.

**M. Barrachin:** Melt contamination with crucible materials is a matter of concern. We should be sure that this will be avoided in the future tests. Otherwise, the matrix of tests with ternary systems must be reconsidered.

**9.Problems and perspectives of SEM/EDX and chemical analysis of corium samples - S.A. Vitol**

In his presentation, S. Vitol talked about the methods of samples elemental analysis employed in PRECOS. He specially dwelt on the XRF and chemical analyses and compared the results obtained during the tests with UO2 – SiO2 and UO2-CaO.

The author also reported that chemical analysis was used as the most reliable one for plotting the liquidus line, and that accuracy of various analytical methods depends on the quality of samples preparation; especially it is true for the SEM/EDX analysis. Also, he told about the difficulties of samples preparation, conducting analyses and solving the these problems.

*Questions.*

**S. Hellmann:** What is repolishing?

**S. Vitol:** The polished sample undergoes polishing another time and the conducting layer is sprayed on it.

**S. Hellmann:** Does repolishing improve the results? Has the problem with the CaO chemical analysis been solved?

**S. Vitol:** Samples repolishing significantly improves the SEM/EDХ results. So far, a search for and elaboration of a chemical technique for the CaO content determination hasn’t been an issue because of the good agreement between the results of chemical analysis (with negligible influence of Ca on the U determination, and with the CaO determination from the residue) and the XRF data (Ca and U determination with the oxygen correction).

**S. Hellmann:** I think I can help to obtain a suitable technique and will put you in touch with my colleague working in this area. I’m happy that the problem with complete corium samples dissolution had been solved.

**P. Piluso:** I have a comment. PRECOS has yielded many new points on the diagram, but it is very important that all the points have been generated using one and the same method, e.g., SEM/EDX. There exist significant differences between three analytical methods, that is, chemical analysis, XRF and SEM/EDX. I should explain it. The XRF is the worst method, as it yields only the cationic composition and gives no idea about the variable valence (Fe oxides may be given as an example). At CEA, we have made a comparative evaluation of different methods employed for corium samples study. However, I’m not sure now if I’ll be able to send you this work.

The EDX is the next method in terms of drawbacks, as it deals with a thin surface layer, and if there is any inhomogeneity, which is quite natural, differences are inevitable.

From the point of view of thermodynamics, it is necessary to know the phases coexisting under certain conditions. I regard chemical analysis as a good one for phase diagram studies. The problem of chemical analysis lies in solubility of a sample. It is clear that samples from different systems dissolve differently, as some samples are oxidic and some are metallic. Spectrophotometry is good, but there exist other more accurate methods, for instance, the high-sensitive inductively coupled plasma mass spectrometry (ICP MS). However, if a sample is homogeneous, SEM/EDX is applicable.

**E. Krushinov:** I agree that chemical analysis is the best option; that is why practically all diagram points from CORPHAD have been obtained by chemical analysis. However, SEM is necessary for plotting tie-lines, as it shows stratification areas and makes it possible to plot points in these areas, and to determine the limits of components solubility. When constructing diagrams, we compare data generated by different methods and make a decision in favour of this or that method only after a very thorough data analysis.

**P. Piluso:** If such a comparison in the course of construction will show a difference of 20-50 rel.%, this will raise many questions. You’ll be told that it is necessary to choose the right method.

**S. Vitol:** You should realize that ICP MS is good only for identifying components present in samples in microquantities. The main component of our experimental samples (uranium) contaminates the instrument, both its sample preparation and introduction unit, and the receiving unit. Certainly, those who can do this analysis feel reluctant to work with our samples. Of course, we know other analytical methods, including the conductometric, emission and atomic absorption ones, but at present we have neither relevant specialists, nor equipment.

**P. Piluso:** The method allows pp, ppm and trace quantities determination, though it’s been designed for a different purpose. The inductive plasma does not require calibration.

**S. Vitol:** This is not so, as all the mentioned methods are relative. Even the declaratively reference-free method of ICP MS requires inner standards. Besides, it is hard telling how the accuracy of the analysis will be influenced by the medium in which samples are introduced into the instrument (mixed concentrated acids).

**G. Langrock:** Reliable results cannot be obtained by one method; they should be compared. Slide 11: it is written there that calibrations were based on chemical analysis. How is it possible?

**S. Vitol:** The XRF calibration for analyzing certain substances uses the calibration specimens or references which can be analyzed by some or several alternative methods, or using certified techniques on standard instruments for physicochemical analysis. Unfortunately, so far we have not found in the Russian Federation any certified techniques for corium composition determination, or standard specimens. Therefore, we used our own fused samples which had been thoroughly studied by chemical analysis. By the way, chemical analysis is among the main methods employed by the passport issuing procedure for the State Standard Specimens (SSS).

**B. Kalinin:** The number of applied methods should not be reduced to just one due to the threat of misinterpretation of the obtained data. The XRF not only allows the elemental content quantification, but also allows quantification of ions of the elements with variable valence, uranium and iron included. The substance analysis of this kind also addresses the thin layer problem.

**P. Piluso:** Both the XRF and EDX measure fluorescence.

**B. Kalinin:** Yes.

**V. Almjashev** shows slides from his presentation and attempts to answer the previously asked questions.

*There followed a discussion targeted more on ‘what we are measuring’ rather than on ‘how well we are measuring’.*

**D. Bottomley:** Was oxygen measured?

**V. Almjashev:** Boron and oxygen were measured.

**D. Bottomley:** What is the problem concerning repolishing?

**V. Almjashev:** The matter is that initially it was very roughly polished, that is the surface was practically unprepared for the analysis. The polishing was accompanied by material crumbling.

**P. Piluso:** What was the analyzed area size?

**V. Almjashev:** A 4 x 4 mm area was analyzed, with a replication for a 20 x 20 µm area. I will show you two documents concerning these analyses.

**P. Piluso:** If you analyzed a 4 x 4 mm area in presence of porosity, there was room for a big error. There should be a certainty that there are no pores filled with epoxy.

**V. Almjashev:** We make a bulk analysis of samples with a quite high degree of porosity, and take an area free of porosity for comparison.

The SEM/EDX analysis is important for the inhomogeneous samples. The difference of results is a good characteristic feature. It refers to the surface preparation for SEM examination. (The results of B4C and standard В2О3 are presented). In this case, surface quality is of high importance. The polished sections for the UO2-CaO system were the worst.

Physical fundamentals of the analysis were presented by V. Knauf. A transition from the analysis quality to representativity of a sample. Typical surface reference specimens and standardization at all stages are required. It should be kept in mind that a survey combined with calibration is a much longer and complicated procedure than the routine analysis. This method should be used rationally, i.e., when doubts about correctness of measurements appear.

**10. Round-Robin SEM-EDS analyses of a corium sample from the Vulcano UB-V6 experiment - P. Piluso**

In his presentation P. Piluso told about analysis certification by several laboratories, namely CEA, ITU and UACH, using one and the same experimental sample. U, Zr, Ca, Si, Mg, C and O were analyzed by SEM/EDX. Convergence of the results for the main elements was quite good, with significant discrepancies noted for oxygen.

P. Piluso thanked D. Bottomley for his assistance with presentation preparation.

*No questions followed.*

**11. Plans for 9-12 quarters - V. Almjashev**

The author presented the experimental matrix corresponding to the Work Plan, told about experimental research planned for the coming year and proposed to cancel investigations of the low-priority systems in favour of the higher priority ones.

**S. Bechta:** Unfortunately we’ve run out of time for a detailed discussion of this proposal.

**S. Hellmann:** We agree to cancel investigations of the eutectic points in the systems **ZrO2–FeO–SiO2 and ZrO2–FeO–CaO** in favour of a more thorough investigation of the higher priority systems.

**M. Barrachin:**

1. We should be informed about the receipt of the license by IVTAN.

2. Can we offer M. Sheindlin something uranium-free to work on? Can they work on that meanwhile?

**M. Sheindlin:** Yes.

**M. Barrachin:**

3. We are interested in one test with the UO2-SiO2 system. All parties have agreed that in the absence of the license operational matters will be discussed by the Operation Agent, and the coordinates of the point will be agreed on after the receipt of the license.

4. Re. Task 3: in case the contamination with Mo will not be overcome, the tests must be terminated and we should discuss this problem; otherwise, we do not need such results.

**V. Almjashev:** Моlybdenum concentrations are insignificant.

**M. Barrachin:** Several %?

**V. Almjashev:** No, the content is below 1%; the problem relates to the iron shift; oxidic mixtures do not contain any Mo. It is not Mo that transits into Fe, but on the contrary Fe transits into Mo.

**Action (6) The Operation Agent will provide collaborators with the data on melt contamination with the crucible material in the already performed tests in UO2-FeO-CaO and UO2-FeO-SiO2 systems. Collaborators will come to a decision concerning reasonability of continuing tests in Mo crucibles.**

**S. Bechta:** Any other questions? I propose to skip the publications item and discuss it later on via email.

In conclusion, S. Bechta thanked all participants for their active contribution to the meeting, expressed appreciation to the collaborators for their attention to the work, and thanked the organizers for preparation of the meeting.

As usual, the next meeting will be held in about a year, in conjunction with the METCOR-P meeting. The time and place will be specified via email.

Project manager Collaborators

S. Bechta M. Barrachin

D. Bottomley

S. Hellmann

M. Fisher

P. Piluso