T2.1.2 Procedures for pre-selection of appropriate methods for the transfer of artefacts between vacuum/inert gas/air

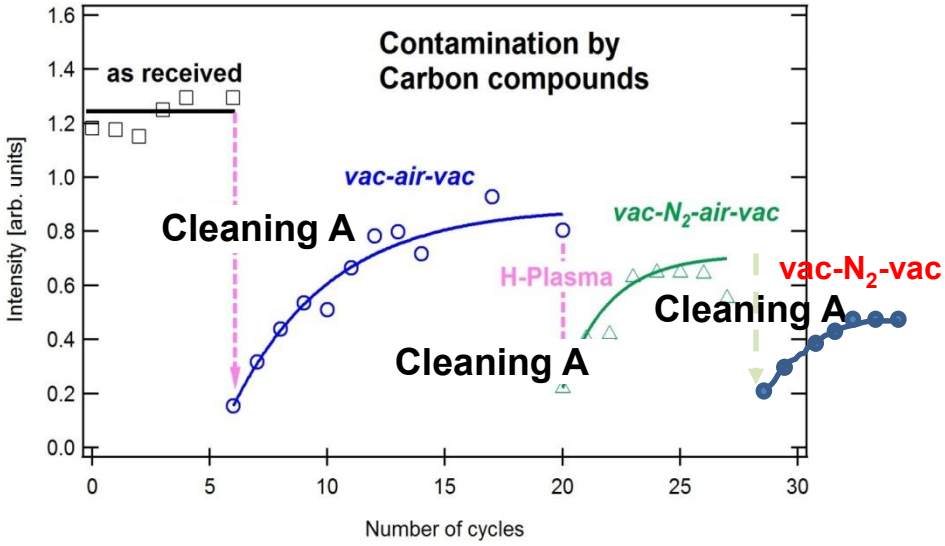
1. Samples

The samples have been cleaned and characterized initially by XPS at METAS. Each laboratory will receive 3 samples of each material they agreed to analyze.

2. Surface analysis by participants

The analysis techniques used are very different. Therefore a precise code of practice can not be provided by the WP leader. The goal is to identify the recontamination in dependence of the processes applied to the different materials.

Preliminary measurements (see graph below) have shown that the contamination on the samples as received is saturated (first 6 cycles):



As discussed and agreed during the SIB-05 meeting on 19th march in Dubrovnik, the workplan needs to be changed as follows:

Sequence of cycles A

1. Initial analysis and cycling **air-vacuum-air** of the batch A of samples (one piece of each material)
2. **Cleaning** by method A
3. Cycling between **air and vacuum** and analysis either at ambient or under vacuum
4. **Cleaning** by method A
5. Cycling between **vacuum-N2-air-vacuum** (starting at ambient air) and analysis either at ambient or under vacuum
6. **Cleaning** by method A
7. Cycling between **vacuum-N2-vacuum** and analysis either at ambient or under vacuum

Sequence of cycles B

Repeat steps 1 to 7 with batch B of samples and cleaning method B

If applicable surface analysis can be performed after each individual step at ambient pressure Nitrogen / air, during the transition and in vacuum, and back. If the changes of one cycle are very small, also multiple cycles can be applied between individual analyses.

For the transfer-process slowly venting using a leak valve is recommended: 20 minutes to 1 hour from vacuum to air. Flushing must be avoided.
The contamination with C and O compounds at ambient is a very fast process. Since we are only interested in differences between different materials and processes, a stabilization time of 5 min. is sufficient before the start of the analysis**. In order to make the measurements comparable apply the same time intervals to all samples and processes. Apply the same storage conditions to all samples.**
For analysis methods where the contamination is removed (eg. TDS) the cycles are:
1 transfer, analysis, 2 additional transfers, analysis, 3 additional transfers, analysis

The vacuum level needed for Watt Balances is 0.5 to $1x10^{-4}$ Pa. However, for some analysis techniques the vacuum level is given by the analysis itself (e.g. XPS, XRR, XRF).

Goal of the cycling experiments is to find a correlation/ dependence between initial surface condition (cleaning) and recontamination

cleaning	**Sequence of cycles (left to right)**		
	vac-air-vac	vac-N2-air-vac	vac-N2-vac
clenaing A	PtIr, AuPt,…Si	PtIr, AuPt,…Si	PtIr, AuPt,…Si
cleaning B	PtIr, AuPt,…Si	PtIr, AuPt,…Si	PtIr, AuPt,…Si
cleaning C	PtIr, AuPt,…Si	PtIr, AuPt,…Si	PtIr, AuPt,…Si

Allocation of cleaning techniques

The table below shows the potential cleaning procedures at different MNI's. The targeted program is cycling after 1 to 3 different cleaning procedures per NMI, if available and applicable. The cleaning methods and parameters for cleaning are:

UV/Ozone: ambient pressure air or oxygen, cleaning time 1 h

H-plasma: 0.7 mbar cleaning time: 30 min

Nettoyage-lavage: rubbing with ethanol soaked chamois followed by steam bath

Others: ultrasonic bath, solvents; soxhlett

	UV/ozone	H-plasma	Nett.-lavage	Others (soxhlett)
NPL	x	x	x	
METAS	x	x	x	
MIKES	x	x	x	
CMI		x	x	
TUBITAK		x	x	x
PTB			x	x
CNAM			x	
MHEST		x		

3. Addional characterization

Non destructive tests (not within WP3) can be performed before or after analysis of WP 3, using the samples of WP3. Some analysis of WP3 can be used for other WP's (WP 1, 4).

Destructive tests should not be carried out with samples from WP3. For destructive tests (e.g. indentation of coated samples deliverable 1.2.2, 1.2.3) METAS can provide additional samples. Please let me know how many and what kind of samples you need for destructive testing.

4. Post-clean storage methods for Task 4.2

The aim of this task is to identify optimum cleaning and post-clean storage methods for each material and produce procedures outlining the cleaning and storage methods for all materials which will be used for primary mass standards identified in WP1 and WP3.

To reduce the workload of each participant I have allocated each participant 3 materials to measure according to the table below:

NPL	EJPD	CNAM	MIKES
Pt/Ir	AuPt	Silicon	Iridium
Silicon	Tungsten	AuPt	Tungsten
Ni alloy	Ni alloy	Iridium	Pt/Ir

Each material is measured by two participants and the materials allocated to the participants are part of the group of materials each participant has agreed to measure for WP3. Therefore no additional samples are needed and participants should use the same samples as WP3 once the measurements for WP3 are complete.

The procedure for the measurements is shown in the table below. Three storage methods are to be tested; Vacuum storage (VS), Air storage (AS) and Nitrogen storage (NS). Each participant should clean the samples using the first cleaning method and then perform surface analysis on the samples. Then the samples are transferred to their storage environments for a period of **1 month** before repeating the surface analysis.

Each participant should perform at least one cleaning method on the samples but it would be an advantage if each participant repeats the process for each of the cleaning methods they will be testing in WP3.

Sample 1	CLEAN A	M	VS	M	CLEAN B	M	VS	M	CLEAN C	M	VS	M
Sample 2	CLEAN A	M	AS	M	CLEAN B	M	AS	M	CLEAN C	M	AS	M
Sample 3	CLEAN A	M	NS	M	CLEAN B	M	NS	M	CLEAN C	M	NS	M

Where:

Clean A, Clean B & Clean C = First, second and third cleaning methods

M = Surface analysis

VS = Vacuum storage (suggested vacuum pressure = 0.1 Pa)

AS = Air storage (suggested pressure = atmospheric + 100 mbar)

NS = Nitrogen storage (suggested pressure = atmospheric + 100 mbar)

5. Report

All parameters should be reported: pressure sample was exposed/ measured, time needed for pumping down and venting time, time of exposure to vacuum, air /N2, details of the cleaning procedure and so on.

Provide a report, comparing the level of contamination (thickness or intensities of contaminants) for different materials and processes as a function of the numbers of cycles.