

**AGREED EUROMET PROJECT
FINAL REPORT**

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| 1. Ref. No.: 734 | 2. Subject Field: mass and related quantities |
| 3. Type of collaboration: cooperation in research | |
| 4A. Partners: BIPM, LNE, LNE-INM, METAS, SP (institutions) | 4B. CEC funded? no |
| 5. Participating countries: CH, FR, SE, (BIPM) | |
| 6. Title: Study of materials for the realization of mass standards | |

7.

Since April 2003, the progress status of this project has been the subject of four short annual reports. A detailed final report is being written. The table below shows the list of all the materials studied between 2003 and 2008.

| Materials (% in mass) | Provider | Density (kg.m ⁻³) | Hardness (Hv) (1) | Magnetic susceptibility (×10 ⁻⁶) (1) | Metallographical structure (1) |
|------------------------------|-----------------------------|----------------------------------|----------------------|--|--|
| Pt-Ir10 | Johnson-Matthey | 21 500 | 175 | +250 | good homogeneity |
| Au-Pt11.4-Ru0.9-In1.5 | Qualident | 19 000 | 180 | -20 | porosity and precipitates |
| Au-Pt10-Zn1 | Qualident | 19 000 | 230 | +15 | - |
| Au-Pt12-Zn1 | Qualident | 19 000 | 210 | +90 | - |
| Au-Pt11-Pd3-Zn2 | Qualident | 18 500 | 225 | +20 | - |
| Au-Pt20-Zn2 | Qualident | 19 000 | 220 | -20 | porosity and precipitates |
| Au-Pt10 | Qualident | | 140 | -23 | - |
| Au-Pt12 | CECM | | 70 | | dendritic cells |
| Au-Pt29 | PXGroup | 19900 | 130 | -16 | porosity and heterogeneity |
| Au-Pt15-B0.1 | PXGroup | 19600 | 115 | -32 | good homogeneity |
| Au-Pt15-Ga3 | PXGroup | 18700 | 160 | -35 | demixing Au and Pt |
| Au-Pt15-Ti1 | PXGroup | 19000 | 75 | -30 | demixing Au and Pt porosity and heterogeneity |
| Au-Ag12-Cu9-Pt4 | PXGroup | 16000 | 210 | -29 | good homogeneity |
| Au-Ag9-Cu12-Pt4 (3) | PXGroup | 16000 | 250 | -28 | good homogeneity |
| Ir (3) | Johnson-Matthey | 22 600 | 385 | +37 (2) | some dislocations |
| Pt-BMG (3) (4) | Liquidmetal Technologies | 15200 | 430 | -17 | amorphous structure (study in progress) |

- (1) experimental value
- (2) bibliographical value
- (3) surface behaviour study in progress
- (4) BMG : Bulk Metallic Glass

Pt-Ir10 alloy is used as reference material for this project, its characteristics are not convenient to be used for the watt balance experiments. The quaternary gold-based alloy (Au-Ag9-Cu12-Pt4) and the amorphous platinum-based alloy (Pt-BMG) of which their surface behaviour study is in progress seem to be the best interesting candidates for the realisation of the transfer mass for the WB experiments. Pure iridium is also interesting but it presents two disadvantages for the WB application. Firstly, it is only available in laminated form, this involves grain deformations along the laminating direction and some dislocations of structure. Secondly, its magnetic susceptibility is positive indicating a weak paramagnetism which is not very suitable for the WB experiments.

In 2007, only LNE and LNE-INM carried on a notable activity in the scope of this project. Their works consisted in preparing artifacts made from four materials, including Pt-10%Ir alloy that acts as the reference material, a quaternary gold-based alloy (Au75%-Ag9%-Pt4%-Cu12%), a platinum-based alloy in an amorphous state, and pure laminated iridium. These artifacts will be used in 2008 :

- to analyze their residual surface contamination by specific techniques such as XPS, ToF SIMS or TDS in order to determine the best cleaning methods and storage conditions.
- to study their mass stability by gravimetric method when transferring them between air, inert media and vacuum (except for amorphous platinum).

Preparation of artifacts for a gravimetric study of mass changes when transferring them between air or inert media and vacuum :

To complete the two 1 kg sets of 5 discs realised in 2006 (one made of Pt-10%Ir and the other made of pure iridium), a third 1 kg set of 5 discs made of quaternary gold-based alloy has been machined, polished and adjusted in 2007. In addition, two travelling boxes have been developed for transporting in each of them a 1 kg cylindrical mass standard made of Pt-10%Ir (one permanently maintained in inert media or air and the other one kept in inert media or under vacuum).

Two 500 g cylindrical mass standards and a stack of 8x125g disks made of Pt-10%Ir have been delivered by BIPM, they will be used in the gravimetric study.

Surface analysis and characterization of selected materials

Preparation of artifacts

Small samples (disks of 10 mm diameter and 1 mm height) made of Pt-10%Ir, of quaternary gold-based alloy and of amorphous platinum alloy have been machined and polished following two procedures and cleaned by using four different methods. They will be studied by XPS, ToF-SIMS and particles accelerator to determine their surface contamination. In particular, it will be examined if there are corrosion and oxidation effects of silver and copper concerning quaternary gold-based alloy.

Preliminary study on pure iridium by thermal desorption mass spectrometry (TDS) :

A 100g artefact made of pure iridium and another made of Pt-10%Ir alloy having a large surface of about 240 cm² and 190 cm² respectively have been realised. After storage in air, we analyse surface contamination molecules during desorption under vacuum up to 600 °C with the TDS apparatus. During the first thermal desorption, water, cleaning solvents and organic molecules up to 45 uma are detected. During the second one, only air components are detected. The surface mass changes in air have been determined after applying a surface desorption under vacuum either at ambient temperature or at 600 °C.

The Joint Research Project “Kilogram – mise en pratique” in which the project 734 could be integrated has not been agreed. We have decided to stop this present Euromet project by the end of 2007, however the French project is going on in 2008. During the next meeting Euramet for Mass and related quantities in March, the possibility to include the future works concerning the subject of the present project in a new European project should be discussed.

8. Coordinator’s name: **Patrick PINOT**
 Address: **LNE-INM/CNAM 292, rue Saint-Martin F-75003 PARIS**
 Telephone: **+33 (0)1 58 80 85 63** Telefax: **+33 (0)1 42 71 37 36** E-mail: **patrick.pinot@cnam.fr**

9. Coordinator’s signature:
signed Pinot

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11. Starting date:
April 2003