BUREAU INTERNATIONAL DES POIDS ET MESURES

LE NETTOYAGE-LAVAGE DES PROTOTYPES DU KILOGRAMME AU BIPM

THE WASHING AND CLEANING OF KILOGRAM PROTOTYPES AT THE BIPM

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La procédure de nettoyage et de lavage des prototypes du kilogramme en platine iridié utilisée au Bureau international des poids et mesures

The procedure for cleaning and washing platinum-iridum kilogram prototypes used at the Bureau International des Poids et Mesures

THE PROCEDURE FOR CLEANING AND WASHING PLATINUM-IRIDIUM KILOGRAM PROTOTYPES USED AT THE BUREAU INTERNATIONAL DES POIDS ET MESURES

by G. GIRARD

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1. **Introduction:** Cleaning of the international prototype, 1882-1946.

> *"The unit of mass is the kilogram. The kilogram is the mass of the international prototype of the kilogram."*

As this definition is rather succinct and says nothing about the physical state of the international prototype, it is interesting to see how it has been interpreted in practice. The international prototype was first used in 1888; then in 1939 and again in 1946 for the 2nd periodic verification of national kilogram prototypes; and, most recently, in 1988-1989 for the 3rd periodic verification.

At the outset, between 1882 and 1889, the first 40 prototypes fabricated from the Johnson-Matthey alloy were compared among themselves in numerous combinations. Each was then compared separately with the international prototype. Before their use in the definitive studies, the prototypes were washed in ethanol vapour and steam. They were then dried under a bell jar in the presence of anhydrous potassium hydroxide. The washing was accomplished with the help of the apparatus shown in figure 1. Note that this device could be used to subject a prototype to jets of either ethanol vapour or steam.

After 1889, whenever the need to compare prototypes arose, preliminary washing was no longer used. Instead, either simple dusting or, in some cases, wiping with solvent took the place of the original washing procedure. We do not know why washing was abandoned. Thus matters stood until 1939, at the start of the 2nd periodic verification of national prototypes. The 1st verification of national prototypes had taken place between 1899 and 1910. The international prototype had not been used and only a small number of other prototypes had been involved.

In 1939, the prototypes were cleaned by rubbing all surfaces with a chamois leather which had first been soaked in ethanol and then in redistilled petrol. Although mass comparisons were interrupted by the advent of World War II, some unexpected results had already been obtained. The wartime period provided an opportunity to review the question of cleaning mass standards as well as to carry out a study of the original washing procedure. The result of this work was the

development of a technique combining solvent cleaning with steam washing.[1] It could be seen from numerous comparisons involving several prototypes that, even though solvents such as benzene and ethanol seemed to clean foreign matter from the surface of a prototype efficiently, such cleaning alone was still incomplete. A final washing with steam was found necessary to remove solvent residues. It was, then, at this time that the need to reinstate steam washing after solvent cleaning was acknowledged. It will be convenient in what follows to refer in general to the procedure combining solvent cleaning followed by steam washing as a "cleaning and washing."

After the war, in 1946, the 2nd verification of national prototypes was once more taken up. The international prototype again was compared with its official copies ("témoins") and with the BIPM working standards after they had all been cleaned and washed. In the following years, all prototypes returned to the BIPM for verification have undergone this same cleaning and washing. Redistilled petrol was replaced by benzene which, because of its high toxicity, was itself abandoned several years ago. Solvent cleaning is now done solely by means of a mixture of equal parts ethanol and ether.

2. Description of present-day procedures.

The treatment of solvent cleaning and steam washing now used at the BIPM is as follows. For cleaning, we use chamois leather which has been previously soaked for 48 hours in a mixture of equal parts ethanol and ether after which the absorbed solvent is wrung out of the leather. This preliminary step removes impurities which might otherwise be deposited on the standards. A second and, indeed, a third soaking are then required to clean the leather sufficiently. When the time comes to clean the standards, each is rubbed fairly hard by hand (we estimate the pressure applied to be of order 10 kPa) over its entire surface using clean chamois leather which has been saturated with the ethanol-ether mixture.

The steam washing which follows the solvent cleaning is carried out using the device shown in figure 2. A round-bottomed Pyrex flask (B) of 1-litre capacity is filled 3/4 full with doubly distilled water and heated by an electric mantle operating at 350 W. As the water boils, the resulting steam passes through a tube which terminates in a small orifice (about 2-mm diameter) directed toward the mass standard (E). The standard is placed on a disk (D) of platinum-iridium which is accommodated by a shallow bowl (C) at the top of a tripod whose upper part can both turn about a vertical axis and be displaced vertically by several centimetres.

With water in the flask boiling away at about 0.5 l/h, the steam jet is first pointed toward the upper surface of the standard, which can be rotated about the vertical axis. The jet is successively directed to all parts of this surface. After a few minutes, the jet is then swept about the cylindrical surface; actually, the jet remains stationary while the prototype is rotated and displaced vertically. The tapered glass tube is maintained at a distance of about 5 mm from the surface of the prototype throughout these operations.

The washing of the cylindrical surface is stopped after about 15 to 20 minutes. Water which has condensed on the surface of the prototype and which has not run off is then absorbed by filter paper. For this operation, an edge of the paper is put

in contact with each drop and the water allowed to flow into the paper by capillary action. Alternatively, the water can simply be blown away with a jet of clean gas.

The standard is then inverted so that it now rests on the base which has just been washed. The washing procedure is continued at the upper surface and ends by a second washing of the cylindrical surface. At present, at the BIPM, the time spent in washing a prototype using the above procedure is about 50 minutes. Needless to say, the disk (D) upon which it rests is previously cleaned and washed by the same method used for a mass standard.

The prototype is then stored on its support beneath a bell jar. No chemical dessicant is used.

Figures 3-12 illustrate the various steps involved in cleaning and washing.

3. Effect of cleaning and washing.

For at least 25 years, all prototypes coming to the BIPM for verification have been compared with the BIPM's working standards, then cleaned and washed, and finally compared again with the working standards. But it was not until 1973, when comparisons began using the NBS-2 balance, that we were able to determine to within a few micrograms (and even more precisely at present) the effect of this treatment. The loss in mass after cleaning and washing as a function of the number of years since the previous treatment is indicated in figure 13. This effect is, of course, also a function of the conditions in which the standard is stored and of the state of its surface. Account must also be taken of the use (if frequent) which has been made of the standard because frequent use may, perhaps, increase the effect.

A straight line (slope -1 microgram per year) has been drawn through the points labelled •. This line, it should be noticed, does not pass through the origin. Points +, representing the international prototype and its official copies, seem to follow the line as well. The points labelled o designate prototypes which have a relatively poor surface.

The fact that the line of figure 13 fails to pass through the origin is consistent with the hypothesis that surface contamination is more rapid just after cleaning and washing. Indeed, a study carried out in 1989 on the international prototype and prototypes Nos. 7, 67, and 73 indicated in essence that their mass increases by about 1 microgram per month during the first three or four months after cleaning and washing as shown in figure 14. For this reason, the national prototypes should, upon their return to their respective laboratories, be cleaned and washed again before they are compared to other standards. In this way their reference mass, i.e. that corresponding to the value measured at the BIPM, should be retrieved.

There are two questions that might be posed about the cleaning and washing process. The first concerns the efficacity of the process in removing surface pollution at the microgram level and the second concerns the cumulative effects of multiple cleanings and washings.

Experiments carried out at the BIPM in 1974 on a newly adjusted prototype shed some light on these questions. Two or three initial treatments were necessary

to remove traces of machining lubricant. The whole cleaning and washing process was then repeated several more times. Each time, the pressure with which the chamois leather was applied to the prototype surface was considerably increased above normal levels. Nevertheless, after each treatment the change in mass of the prototype was found to be within the weighing uncertainty.

On the assumption that the effectiveness and reliability of the cleaning and washing process had thus been demonstrated, the international prototype and its official copies were cleaned and washed twice at the start of the 3rd periodic verification of national prototypes. After each cleaning and washing they were compared with two prototypes that served as reference standards. The results are shown in figure 15. A single cleaning and washing appears to remove about 90 % of the surface contamination. Therefore, if a second treatment removes only a few micrograms, we can predict that a third treatment would have no practical effect (note, for instance, the second cleaning and washing of prototype No. 25).

It was on the basis of these results that we decided that national prototypes sent to BIPM for the third periodic verification will receive two cleanings and washings. No mass comparisons will be made between the first and second, however, because the relative unimportance of such data does not justify the considerable time and effort which such a programme would entail.

Reference

[1] BONHOURE, A. *BIPM Proc.-verb. Com. int. poids et mesures,* **20**, 1946, pp. 171-178.

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Figure Captions

1. Apparatus used between 1882 and 1889 for washing platinum-iridium prototypes. Alcohol vapour and steam were directed alternately towards the prototype.

2. Sketch of the apparatus now used for the washing of prototypes. E: prototype; B: 1 litre Pyrex flask containing bi-distilled water; C: bowl for collecting condensed water and allowing it to run off; D: platinum-iridium disk upon which the prototype is placed.

3. Cleaning a prototype by means of chamois leather moistened with a mixture of equal parts ethanol and ether. Cleaning a base.

4. Cleaning a prototype by means of chamois leather moistened with a mixture of equal parts ethanol and ether. Cleaning the cylindrical surface.

5. Washing the platinum-iridium disk (D) placed in the bowl (C).

6. Manipulating a prototype by means of a pincers. The bearing surfaces of the pincers are covered with an inner layer of chamois leather and an outer layer of lens tissue.

7. View of the entire apparatus.

8. Washing the base of a prototype.

9. Washing the cylindrical surface of a prototype.

10. Using a jet of clean gas to eliminate the last drops of condensed water.

11. Setting the prototype upon a Pyrex disk after washing.

12. Storage of the prototype under a bell jar.

13. The change in mass Δm found on cleaning and washing Pt-Ir prototypes of the kilogram plotted as a function of the number of years since the last cleaning and washing. Open circles represent prototypes having very poor surface states. The crosses (+) refer to the international prototype and its official copies.

14. Increase in mass observed after cleaning and washing of the international prototype $\mathfrak{K}$ and Nos. 7, 67 and 73. Points denoted A and B indicate the times at which the mass is, respectively, placed in and removed from the balance; C is a correction applied to the mass of No. 73 to account for additional mass due to its having been kept, uncovered, in a laminar flow chamber. The vertical bars on each data point represent the type A uncertainty of the weighing.

15. The change in mass, Δm, of the international prototype $\mathfrak{K}$, its six copies, and No. 25 on cleaning and washing.

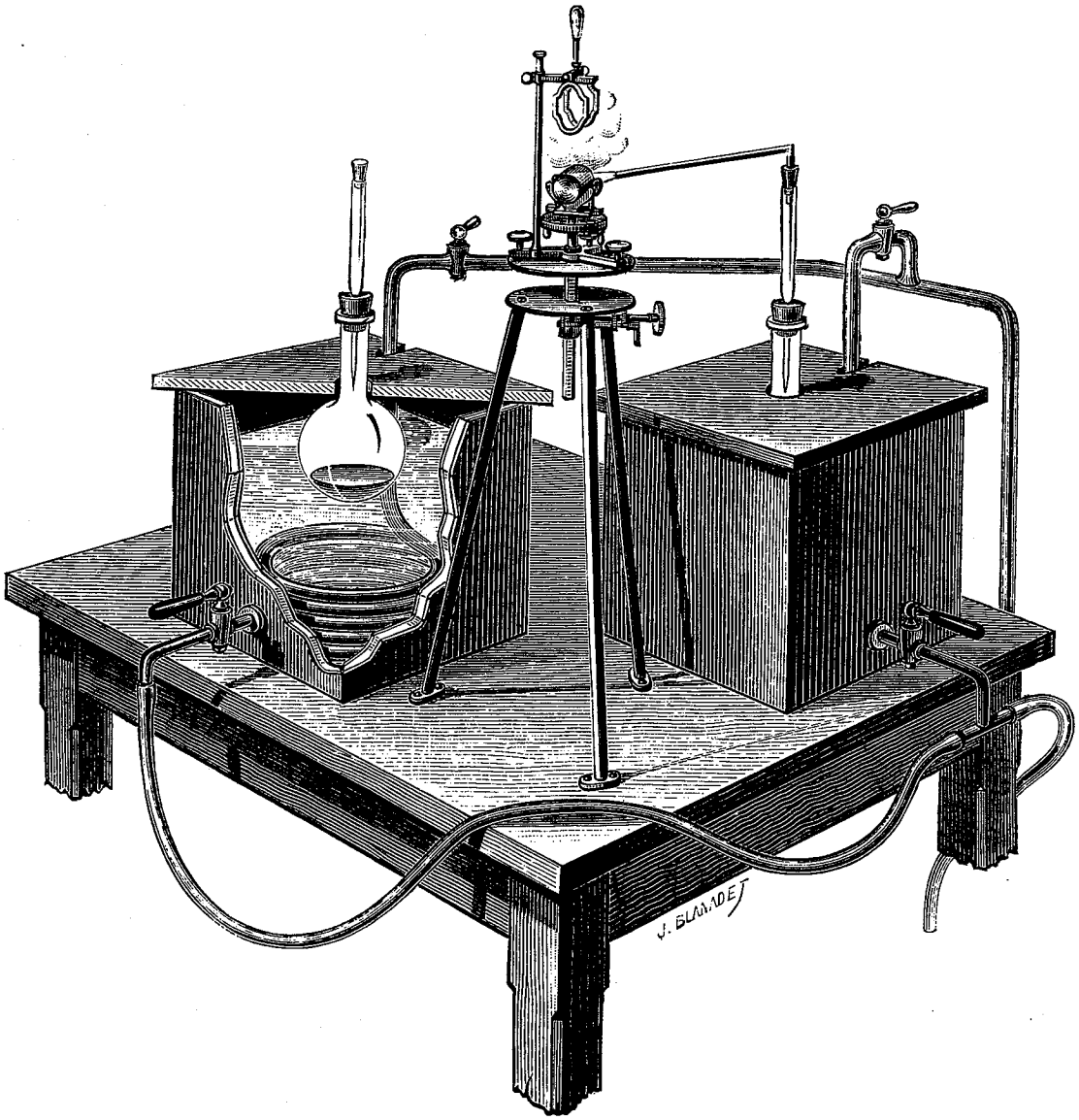


Figure 1

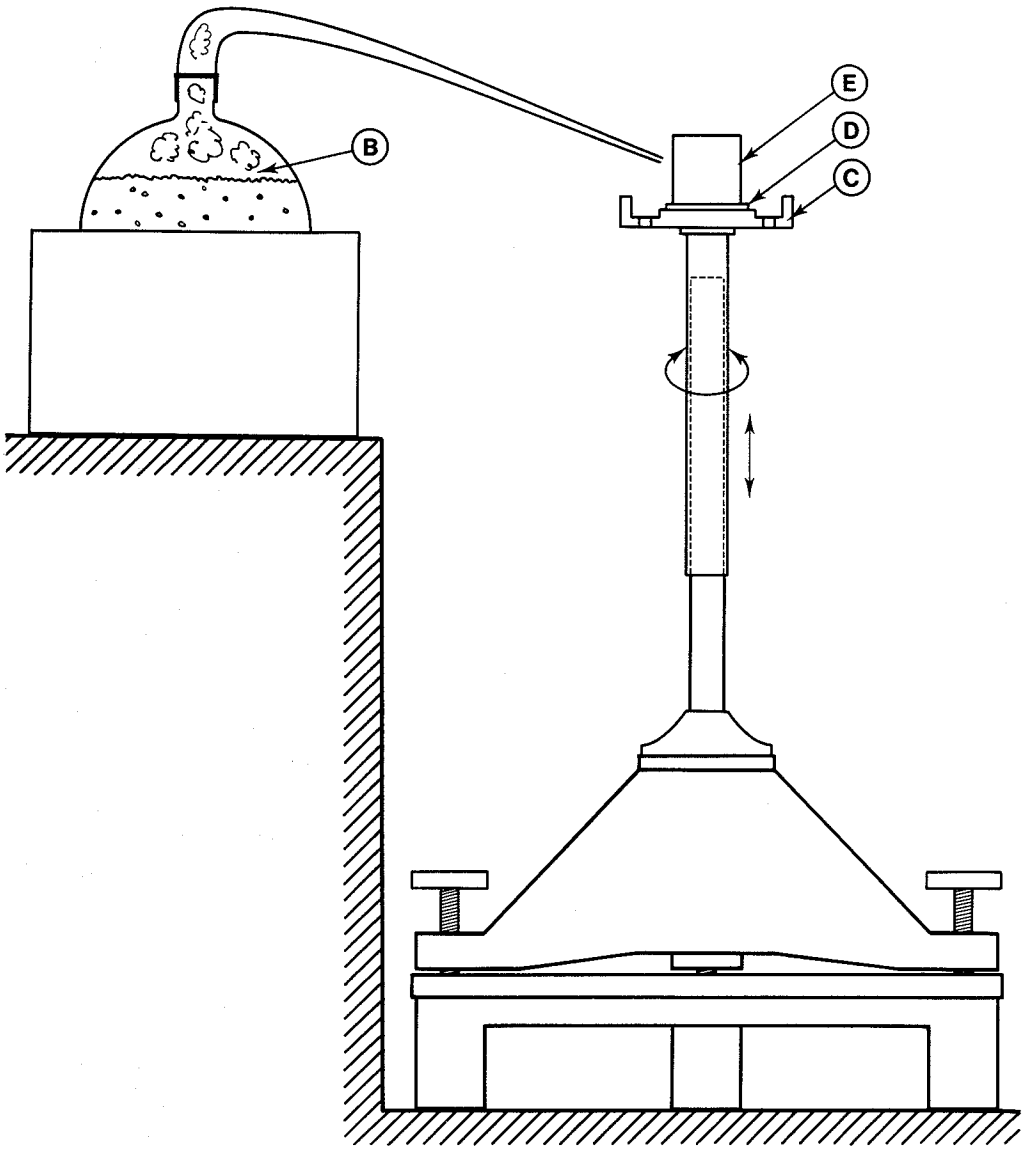


Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

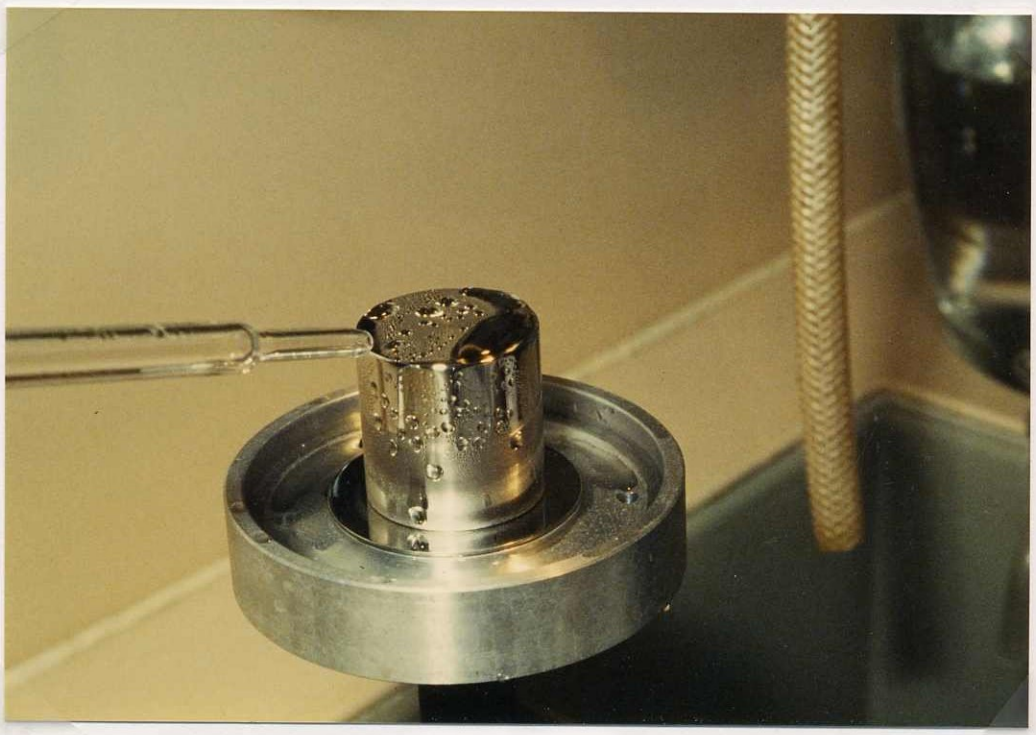


Figure 8

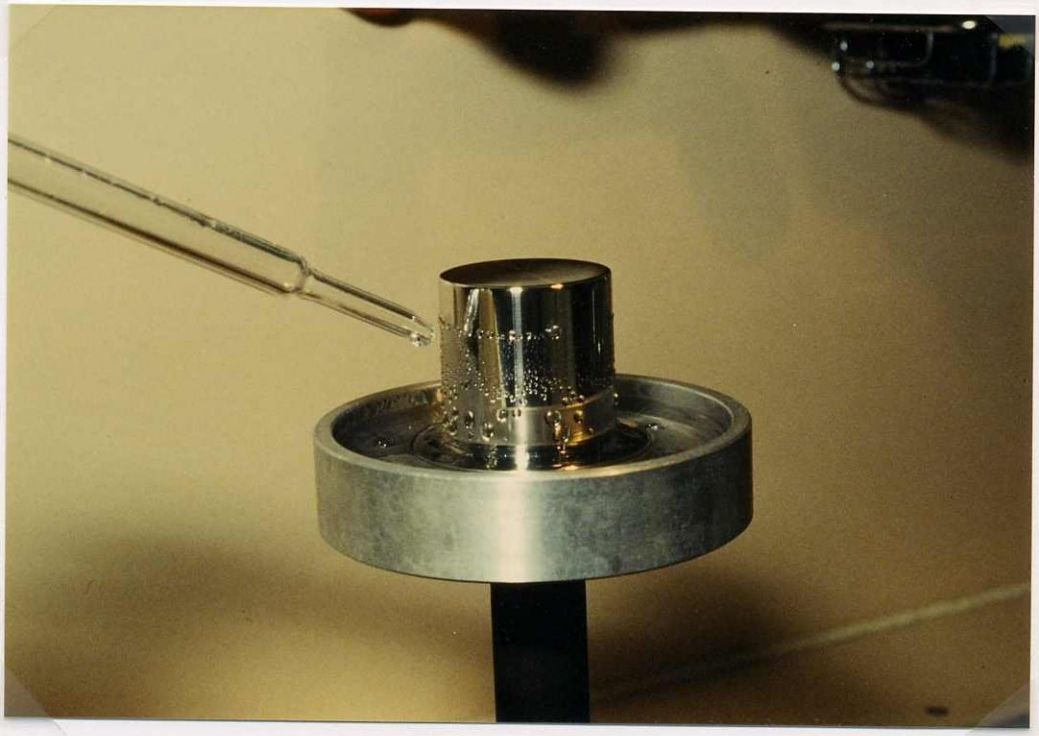


Figure 9

Figure 10

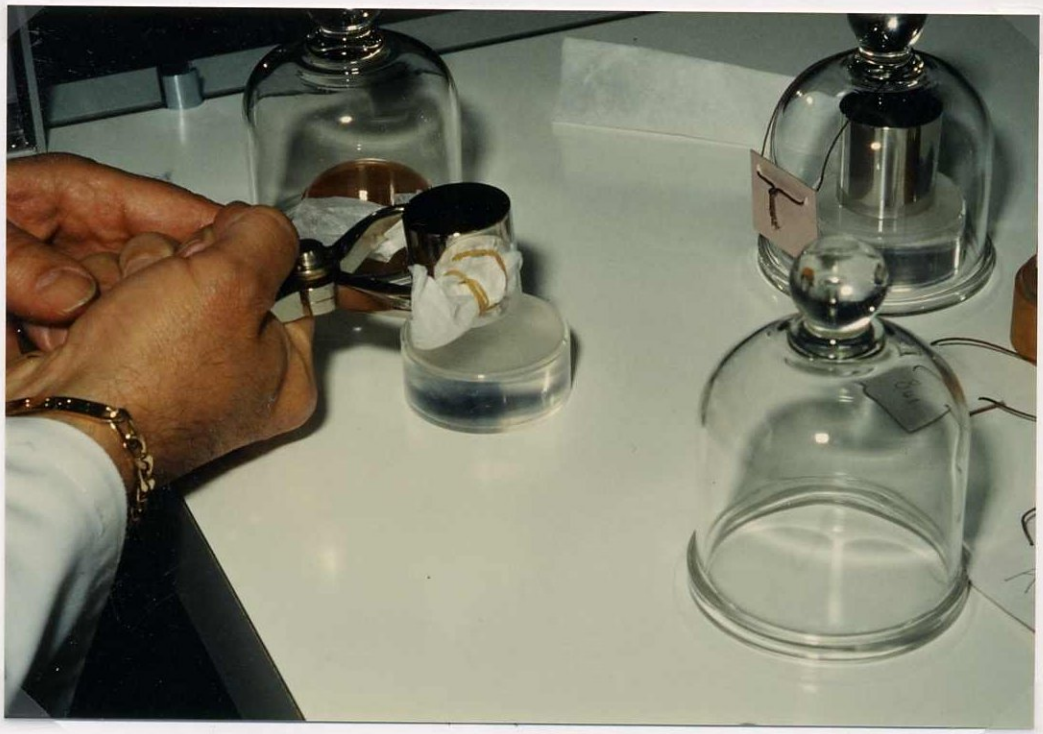


Figure 11

Figure 12

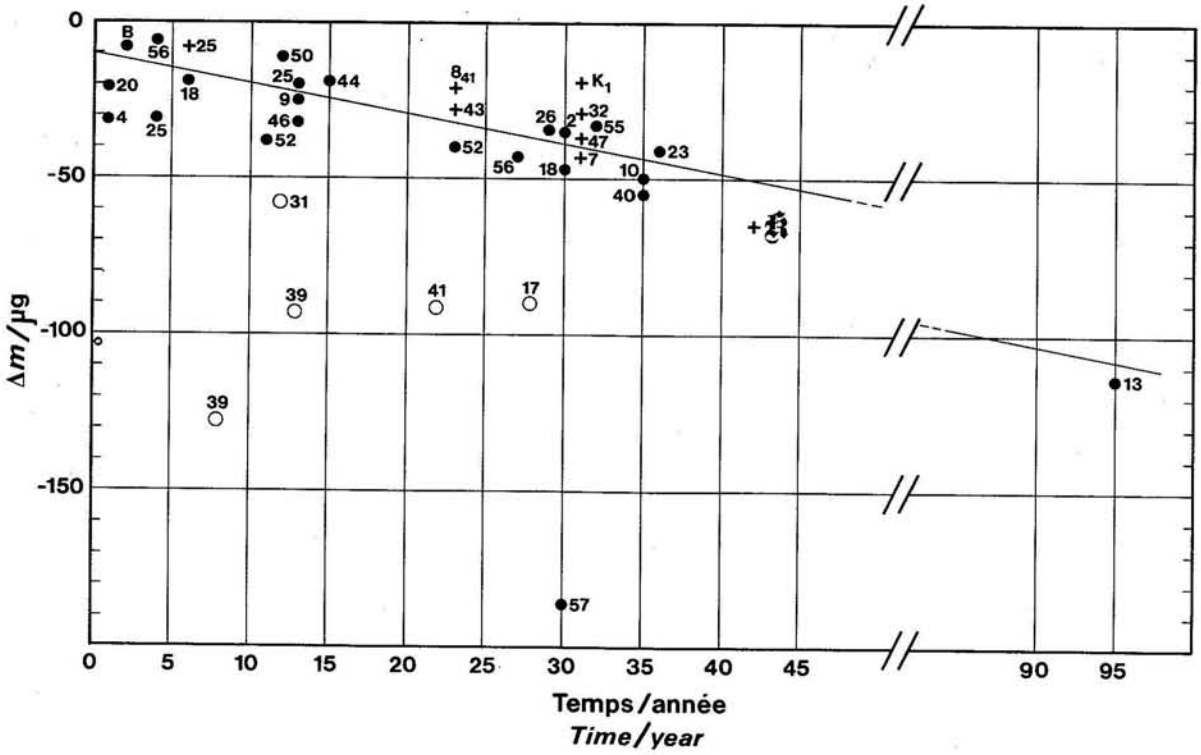


Figure 13

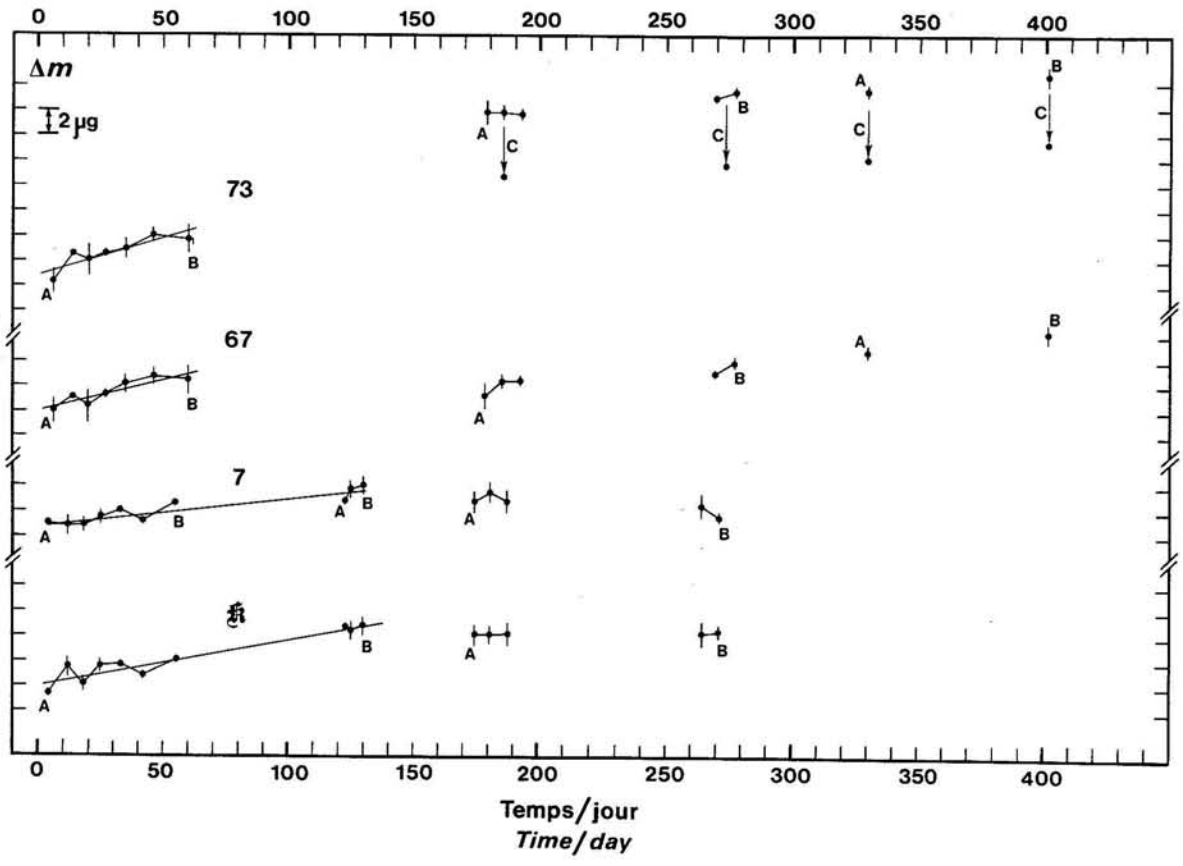
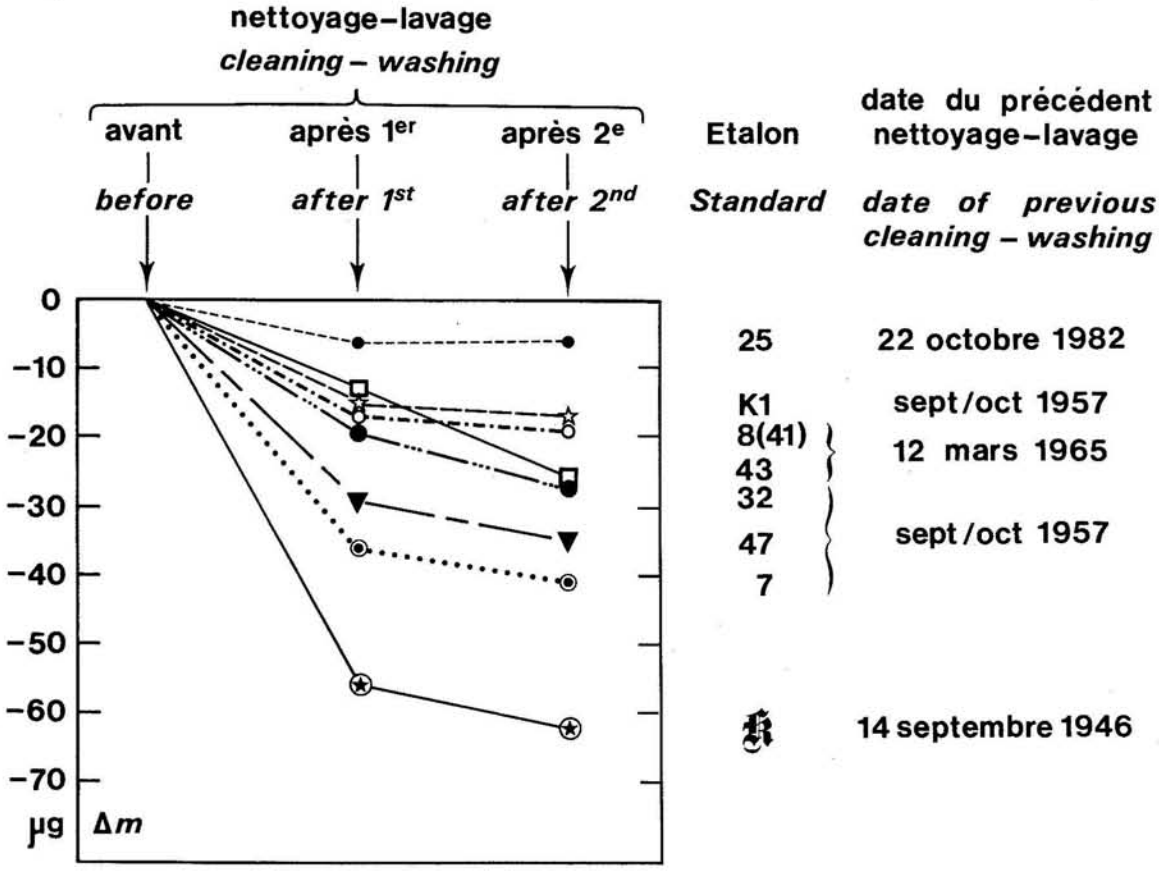


Figure 14


nettoyage–lavage
cleaning – washing
avant
before
après 1er
after 1st
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Etalon
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Figure 15