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AN OVERVIEW OF EXPERIMENTAL STUDIES ON H/PD OVER-LOADING WITH THIN PD WIRES AND DIFFERENT ELECTROLYTIC SOLUTIONS

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Abstract

Hundreds of electrolytic loading tests of thin **Pd** wires in different experimental conditions have been performed in order to find out the best procedures for a stable, high Hydrogen overloading into the Palladium lattice.

In a very diluted acid solution thin **Pd** cathodes (50 or 100 μm in diameter) and tick **Pt** anodes (0.5 mm in diameter) were used in a parallel or coaxial geometry. Normalised resistance (R/R_0) of the **Pd** cathode was on-line and continuously measured in order to assess the actual **H/Pd** values.

Different electrolytic solutions have been tested by adding to the acid solution very low amounts of **Ca**, **Sr**, **Li** and **Hg** ions; high loading **H/Pd** ratios have been achieved with a satisfactory grade of reproducibility.

Several loading procedures have been performed in a wide range of electrolysis current (from a few mA up to one hundred mA) and at different **Hg** ion concentrations.

The obtained results allowed for the definition of a loading protocol capable to insure very high H/Pd over-loading. Stable $R/R_0 \leq 1.2$ values (corresponding to **H/Pd** ratios ≥ 1) can be currently achieved with an extremely low electrolytic power supply (10 V; 5 mA).

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1. INTRODUCTION

During the last 6 years we spent a lot of efforts in order to find out the best Hydrogen loading procedures of thin Pd cathodes.

Most of the researchers agree that, in cold fusion experiments, in order to obtain stable and reproducible excess heat, it is necessary to achieve and maintain very high **D/Pd** (≥ 0.85) loading ratios [Ref. 1]. The poor results generally achieved by the conventional electrolytic techniques, based on the use of **LiOH** solutions, especially from the point of view of their reproducibility, induced us to develop a completely different approach. In fact, in our previous papers, we have reported a reproducible procedure to achieve very high loading ratios using Palladium thin wires (**H/Pd** ≈ 1) [Ref.s 2,3]. This procedure is based on the increasing of the cathodic over-voltage (which is known to be the main controlling parameter of the **H(D)-Pd** loading) by modifying the nature of the cathode surface (i.e. by inducing the formation of a very thin layer of an alkaline-earth carbonate on its surface).

2. EVALUATION OF THE H/PD RATIO

In order to estimate the actual **H/Pd** atomic ratio of the **Pd** cathode during the electrolytic loading process, we measured the electrical normalised resistance (**R/R_o**) of the **Pd** wire; i.e. the ratio between the actual resistance (**R**, during the loading) and that of the electrode at the beginning of the electrolysis (**R_o**), when the value **H/Pd**= 0.

The loading ratio was inferred and continuously monitored by means of the well known relationship [Ref.s 4,5,6] between the resistance and the **H(D)** content in the **Pd** matrix (**Fig. 1**). The actual known values of this relationship terminate at **H/Pd**= 0.9, corresponding to **R/R_o**= 1.40. Beyond such values the correspondence can be estimated only by a linear extrapolation. Accordingly, it is possible to assess that the ratio **H/Pd**= 1.00 is roughly achieved when **R/R_o**=1.20.

3. EXPERIMENTAL APPARATUS

A schematic diagram of the experimental set-up is shown in **Figure 2**. The vessels are typically cylindrical glass beakers of different sizes (from 0.5 up to 5 litres).

The power supply can operate either at constant D.C. current or at constant D.C. voltage. The cathode is grounded. The voltage is applied to the anode through a home-made impedance adapter circuit (impedance booster), in order to avoid a current feed-back from the A.C. measuring circuit. The latter is essentially composed by a pulse generator (allowing for sinusoidal, square and triangular wave forms; we always used the sinusoidal ones) and a ground home-made coupling circuit (ground return, both for the D.C. and A.C. generators).

Temperature sensors (based on Silicon integrated circuit) are located inside the cell (into the solution at 3 different levels) and outside the cell (2 for the thermal bath and 1 for the room temperature) and on-line monitored.

The Pd cathode, because of its favourable surface/volume ratio, allows for a very fast Hydrogen absorption and its high resistance (about 8 Ω) favours both the accuracy and the precision of the measurements. Furthermore, the 1/r dependence of the electric field around the

wire allows for a sharp increase of the pH value just in its proximity, thus promoting the carbonate precipitation in that region only [for more details see: Refs. 2,3].

--- The geometry of the electrolytic cell strongly affects the loading process: the relative position of the electrodes is crucial for a proper set up of the primary electric fields during the electrolysis [Ref.7]. Moreover we have to take into account that during the loading the **Pd** wire's length remarkably increases (5→15%); the wire is consequently forced to bend and the original distance of the electrodes could be significantly changed. We tested 2 different **Pd-Pt** electrodes distance values (variable from 1.5 cm up to 7 cm) in 2 different geometries:

- 1) a parallel geometry with **Pd** and **Pt** wires of the same length at the same distance;
- 2) a **Pd** central axial geometry with 4 **Pt** wires cylindrically located around the cathode at the same distance.

--- The electrodes are about 25 cm long; The **Pt** wire (sector AB as drawn in **Fig. 2**) was 1 mm thick; **Pd** cathodes of 2 different thickness values (50 or 100 μm) were used. A pick-up junction divides the **Pd** wire in 2 equally long sectors (sectors CD and DE named “up” and “down” as drawn in **Fig. 2** of electric resistance about either 2 or 8 Ω respectively for either 100 or 50 μm **Pd** diameters) allowing for the measurement of the resistances of the correspondent wire segments.

--- **H₂O**, **Ethyl Alcohol (C₂H₅OH)** or **Methyl Alcohol (CH₃OH)** were used for the preparation of the electrolyte (in order to reduce the impurities present in commercial heavy water [Refs.8,9] some tests using alcohol based electrolytes were previously performed in the electrolytic loading in **D₂O/C₂H₅OD** solvents; in the present work the alcohol based electrolytes were also tested for Hydrogen loading).

As above reported, very low concentrations (less than 1 mMole) of **HCl** or **H₂SO₄** were added to the electrolytes in order to maintain the pH around 4.5 \pm 5.5; the anode-cathode electrolytic resistance ranged between 1 and 5 K Ω).

--- Small amounts (tenth of μMoles) of different alkaline elements such as **Ca**, **Sr** and **Li** were added to the electrolyte, according to an our original procedure previously developed in order to improve the **H/Pd** loading (motivations and details of this addition are also reported in [Ref.3])

--- Finally, many tests have been performed by adding very small amount of **HgCl₂** (ranging from 0.1 up to 10 μMoles) to the electrolyte. In some tests very small amounts of **Hg** (estimated on the order of tenth of nMole) were actually present in the electrolyte (nominally **Hg⁺⁺** free). In fact, in consequence of the de-loading process of the cathode normally effected at the end of a set of experiments, the **Hg** amalgam previously formed on the Pd surface in the cathodic cycles is stripped away during the final anodic cycle and some residual traces normally remains on the **Pt** surface even after the **Hg** containing electrolyte has been fully removed. **Hg** traces on the **Pt** surface go back into solution as soon as the cathodic cycle of the new set of experiments starts up.

Mercury, as it is shown below plays a fundamental role in the over-loading process.

4. H/Pd LOADING PROCEDURE

In the achievement of very high **H/Pd** loadings, the role played by the electric parameters (Anode-Cathode voltage and current) is crucial with any given cell geometry and electrolyte. The primary and secondary electric field [Ref.7] operating onto the **Pd** cathode may produce an **Hg** and alkaline element containing deposit with a particular structure, which seems to be responsible for the **Pd**-bulk over-loading.

In order to produce this particular structure, we tried several procedures, changing electrolysis current values not only at the electrolysis start up (Start in Low current: **SL** or Start in High current; **SH**) but also during the electrolysis when the **Pd** electrode had been loaded and was steady, after the "peak value" of $R/R_o=1.8$ (as referred at the **Fig. 1**).

Low current (Low: **L**) means just a few milli-Amperes (2→10 mA and 5→15 Volts), high current (High: **H**) means some tens of milli-Amperes (30→150 mA and 50→200 V) and mean current (**M**) is occurring in the middle range.

In the following a list of these loading procedures (depending on the **Hg** deposition onto the **Pd**) is reported:

- **Start and Load**: start at constant current (**Low** or **High**) until the **Pd** cathode reaches a consistent over-loading ($R/R_o < 1.3$); in general, if the **Hg** concentration is high, when the current is switched off, a very slow de-loading occurs (in one test no de-loading was observed along two days) [Ref.10]. We call this condition **H/Pd**-locked.
- **OFF/ON**: if the **Pd** electrode is loaded just over the R/R_o "peak", the current is switched off, allowing for the **Pd** to de-load to the "peak"; the current is then switched on (either at low or at high current); this cycle can be repeated several times until the **Pd** reaches a high loading. This procedure can be applied when the **Hg** concentration is very low and the "ON" condition is corresponding to high current during the previous cycles.
- **L/H**: is similar to OFF/ON, but the current is not switched off, and "OFF" corresponds to a low current while "ON" corresponds to a high current.
- **L/H/L**: is similar to the previous L/H, but after **Pd** achieves high and steady loading at high current condition, current is set in low condition; at this low current a fast de-loading occurs, but sometimes the de-loading stops and **Pd** reloads slowly up to high values (sometimes higher than the ones obtainable at high current). This procedure is effective when **Hg** is neither very diluted nor very concentrated.

5. EXPERIMENTAL TESTS

In **Table 1** are reported only the most revealing tests out of the many hundred ones that were performed. In this table all the above cited parameters are reported, that is: cell geometry, **Pd** thickness, electrolytic solution, electric values, loading procedure. The column "**best R/Ro**" means the final **H/Pd** over-loading reached applying the proper procedure (relative to "up" and "down" **Pd** sectors).

The 20 tests in **Table 1** are in chronological order (from 1998 till 2004) and some of them have been repeated many times; the "best R/R_o " reproducibility was very poor at the beginning (about 10%) and increased with time reaching at present reasonably good values (>50%),

particularly when the **Hg** concentration is finely tuned and an optimal “current cycle” procedure is adopted.

In **Fig.3** and **Fig.4** are reported the values of R/R_o vs time (corresponding to the parameters of **Tab.1,1** and **Tab.1,2**: the first two rows of Table 1). In these figures it is possible to see that alkaline elements like **Ca** and **Sr** are quite equivalent for the achievement of high loadings with a high **Hg** concentration. A typical “**Start and Load**” procedure is also shown. In this case the **Pd** electrode very often appears to be covered with a very thin **Hg** film which is nevertheless so impervious as to explain the observed very slow de-loading process when the current is switched off (load+lock).

In **Fig.5** and **Fig.6** a typical **OFF/ON** operation is shown in connection with runs with **Sr** and “residual” **Hg** (**Tab.1,3** and **Tab.1,4**). It is shown that it is possible to achieve a high and steady loading from low loading ($R/R_o=1.8 \rightarrow 1.2$). Moreover, the de-loading curve (electrolysis in **OFF** for 1 day) shows that the resistance measurements are correct and consistent (peak at $R/R_o=1.8$ and return to $R/R_o=1.0$, i.e. the starting condition); the de-loading vs time curve allows for the observation of the typical $\beta \rightarrow \alpha + \beta$, **Pd-H**, lattice phase transition (occurring at $R/R_o \cong 1.68$, $H/Pd \cong 0.6$ at room temperature and at a pressure of 1 atmosphere [Ref.11]).

In **Fig.7** (run of **Tab.1,7**) it is shown the role played by the **Hg** addition during the run (at the time of about 75 Ksec) in combination with a **Low/High** operation; high loading values persist even when the current is decreased.

The effects of the substitution of **H₂O** with **Ethyl Alcohol** (as base for the electrolyte) are shown in **Fig.8** (run of **Tab.1,11**): the presence of **Sr+Hg** in sulphuric environment is effective for reaching very high loading, by applying a **OFF/High/Low** current procedure.

Even with very small **Hg** additions (down to 0.5 μ Moles), it is possible to reach very high loadings ($R/R_o=1.05$) as shown in **Fig.9** (run of **Tab.1,15**). In this case just a **Start-Low/High** current variation is sufficient to trigger the loading process.

By exchanging **Sr** with **Li** similar high loading effects are obtained (runs of **Tab.1,16** \rightarrow **1,19**) but additions of fair amounts of **Hg** (1 μ Mole), cause the role played by the electrolytic current to become crucial, as shown in **Fig.10** (run of **Tab.1,20**). In this plot, the first run is performed at **Low** current (5 mA) resulting in a poor loading. On the contrary with the subsequent run, which was started with a **Medium** range current of 36 mA (after a de-loading at $R/R_o=1$, with -5 mA anodic current) a steady high loading ($R/R_o=1.2$) was obtained within a few hours.

Several tests with **Methyl Alcohol** have also been performed showing loadings around $R/R_o=1.3$.

We never measured “anomalous” temperature variations inside the cell even when very high loadings were achieved.

Tab.1 – The most relevant tests (out of many hundred ones) showing high over-loadings performed by different solutions, elements addition and loading procedures (tests are been performed at room temperature of about $22 \pm 5^{\circ}\text{C}$).

	1.1.1.1. d ϕ		Alkaline Element	HgCl ₂	Power <i>1.1.1.1.3.1.1 apply</i>	<u>LOAD</u> procedure	“best” <i>R/Ro</i>	<u>Remark</u> <i>(set-up)</i>
1	50	H ₂ O=2400cc +HCl= 140	CaCl ₂ = 70	Hg ≈ 10	(60;20)	Start and Load	(1.28;1.35)	K-A=7 cm geo. parallel
2	50	H ₂ O=2400cc +HCl= 140	SrCl ₂ = 35	Hg ≈ 10	(40;20)	Start and Load	(1.15;1.18) <i>(load+lock)</i>	idem
3	50	H ₂ O=2400cc +HCl= 210	SrCl ₂ = 180	very low (≪ 0.1)	(150;90)		(1.16;1.20)	K-A=6 cm geo. parallel
4	50	H ₂ O=2400cc +HCl= 210	SrCl ₂ = 180	very low (≪ 0.1)	(150;65)	OFF/ON <i>(+ deload)</i>	(1.10;1.11)	idem
5	50	H ₂ O=2400cc +HCl= 500	SrCl ₂ = 20	very low (≪ 0.1)	(100;77)	OFF/ON	(1.20;1.25)	idem
6	50	H ₂ O=2400cc +HCl= 250	SrCl ₂ = 160	very low (≪ 0.1)	(150;70)	OFF/ON	(1.25;1.30)	idem
7	50	H ₂ O=5000cc +HCl= 250	SrSO ₄ = 60	Hg=10cc	(90;105) →(40;50)	LOW/HIGH /LOW	(1.12;1.15)	K- A=1.5cm geom. axial
8	50	H ₂ O=1200cc +HCl= 20	SrCl ₂ = 30	very low (≪ 0.1)	(140;133)	OFF/ON	(1.15;1.15)	idem
9	50	H ₂ O=1200cc +HCl= 20	SrCl ₂ = 30	very low (≪ 0.1)	(6.5;4.4)	HIGH/LOW	(1.28;1.28)	idem
10	100	Ethyl=395cc +H ₂ O=20cc H ₂ SO ₄ = 25 +HCl= 210	SrCO ₃ =17mg <i>(powder)</i>	Hg= 5	(11;2)	Start and Load	(1.25;1.30)	idem
11	100	Ethyl=395cc H ₂ SO ₄ = 25 +HCl= 210	SrCO ₃ =17mg <i>(powder)</i>	Hg= 2.5	(11;2.5)	Start OFF/H/L	(1.15;1.15)	idem
12	100	Ethyl=395cc +H ₂ O=20cc H ₂ SO ₄ = 10 +HCl= 200	SrCO ₃ =30mg <i>(powder)</i>	Hg= 8	(12;3.7) → (12;1)	Start and Load	(1.34;1.34)	idem
13	100	H ₂ O=400cc +HCl=200	SrCO ₃ =85mg <i>(powder)</i>	Hg= 5	(45;60)	Start in L /M/H	(1.30;1.35)	idem
14	100	H ₂ O=400cc +HCl=200	SrCl ₂ (6H ₂ O)= 53mg	Hg= 2.5	(6;7)	Start and Load	(1.33;1.36)	idem
15	50	H ₂ O=420cc +HCl= 70	SrCO ₃ = 7mg <i>(powder)</i>	Hg= 0.5	(10;6) → (50;45)	Start in L /H	(1.07;1.05)	idem

16	50	H ₂ O=420cc +HCl= 70	SrCO ₃ = 7mg (powder)	Hg= 0.5	(10;6)	H/L	(1.31;1.26)	idem
17	50	H ₂ O=420cc +HCl= 70	LiOH= 50	Hg = 0 → 0.2	(10;6) → (48;34)	L/H/L	(1.25;1.25) (1.21 in H)	idem
18	50	H ₂ O=420cc +HCl= 70	LiOH= 50	Hg= 0.3	(10;5) → (48;34)	L/H/L	(1.33;1.31) (1.19 in H)	idem
19	50	H ₂ O=420cc +HCl= 70	LiOH= 50	Hg= 0.1	(10;6) → (49;53)	L/H/L	(1.30;1.30) (1.22 in H)	idem
20	50	H ₂ O=420cc +HCl= 100	LiOH= 50	Hg= 1	(10;5) → (42;36)→	Start in L→ Start in H→	→(1.5;1.5) →(1.2;1.2)	idem

6. DISCUSSION

Taking into account also previous studies (particularly performed during last 2 years), we can confirm that our procedure for the **Pd-H** overloading up to 1:1 loading ratio is effective using both aqueous and alcoholic solutions. This method is based on a proper deposition of a **alkaline+mercury** containing thin film onto the **Pd** cathode surface (independent of the wire section). Particular current cycles can improve the loading.

The complexity of these tests and the large spread of the process parameters distribution, can be tracked down to a peculiar deposition layer (ranging from 20 to 200 nm) onto the **Pd** cathode. We conjecture the formation of a sort of nano-structure on the surface which can give rise to relevant electro-chemical potentials and locally high current densities [Ref.12].

During the performance of a test consisting of a dozen of high current loadings (120 V; 60 mA) at high **Hg** concentration (about 10 μMoles) it was observed that after each loading/de-loading cycle the Ro value increased of some percent. At the end of the test we measured the **Pd** wire thickness and it was found a decrease in diameter from the original 50 to 46 μm. These values are consistent with the increase of Ro with respect to the one at the beginning of the test. It is reasonable to assume that the **alkaline+Hg+Pd** layer formed during the loading (cathodic) cycle issued in the removal of about a 200 nm **Pd** layer during each de-loading (anodic) cycle.

A further structural analysis of this deposition is required to confirm the rightness of our conjecture.

It was shown [Ref.9] that the achievement of high loadings with **D₂O** instead of **H₂O** is much more difficult. Nevertheless we think that our method could be transferred to heavy water solutions. New tests are in progress showing encouraging preliminary results (R/Ro≅ 1.55 at low regime of 5 mA). The obstacles limiting the over-loading of **Pd-D** systems are mainly due to impurities present in the commercial **D₂O**.

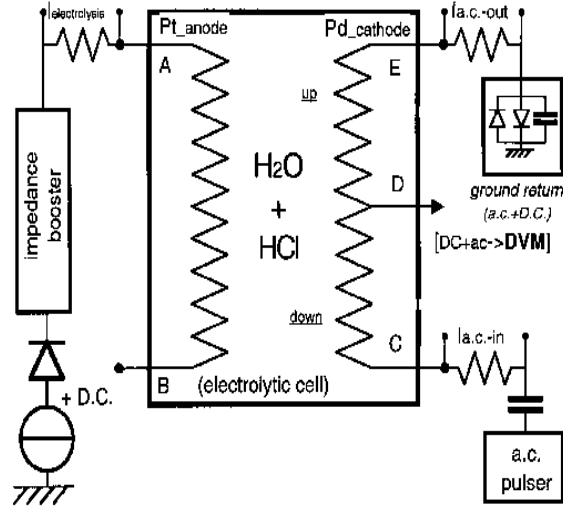
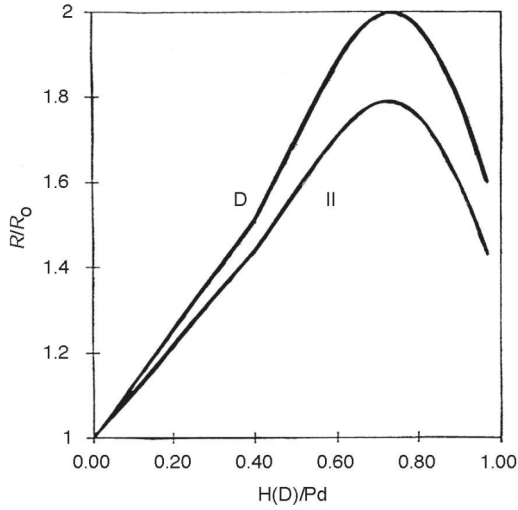


Fig. 1 – Normalized Resistance R/R_0 vs $H(D)/Pd$. **Fig. 2** – Electrolytic cell: a schematic view.

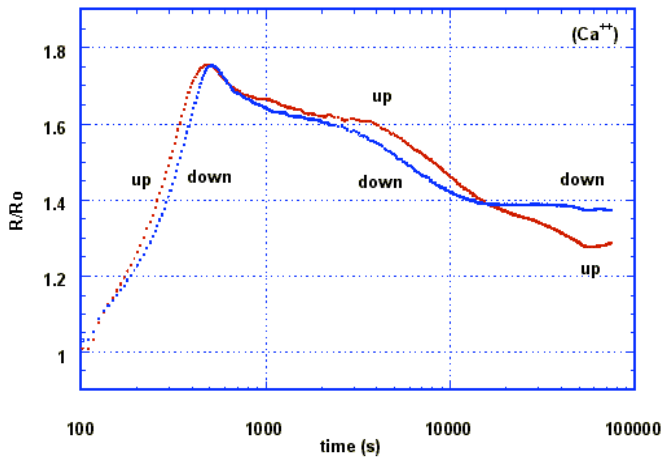


Fig. 3 – Test “Start and Load” with Ca ions and high Hg concentration (Tab.1,1).

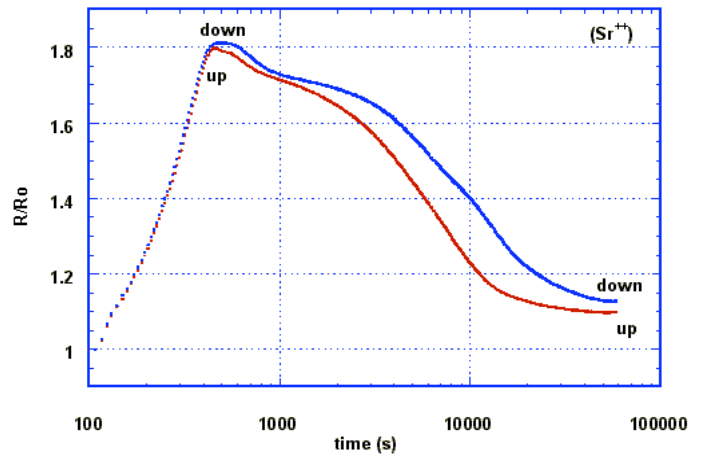


Fig. 4 – Test “Start and Load” with $Sr + Hg$ ($10\mu M$), ending with a H/Pd “lock” (Tab.1,2).

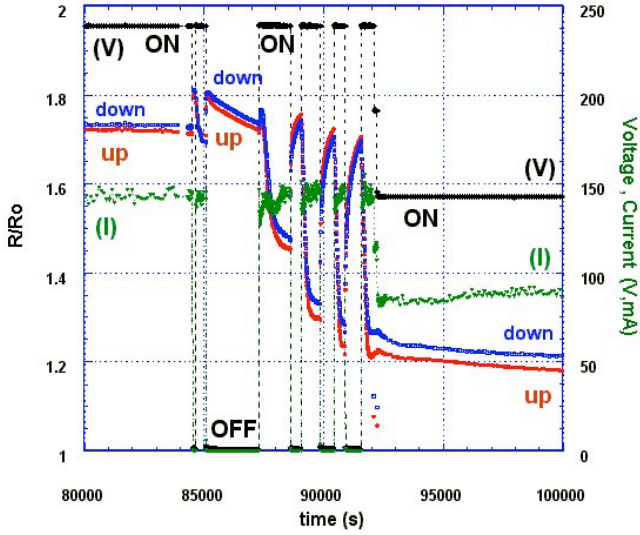


Fig. 5 – Test of OFF/ON procedure with Sr (+ Hg very low concentration) (Tab.1,3).

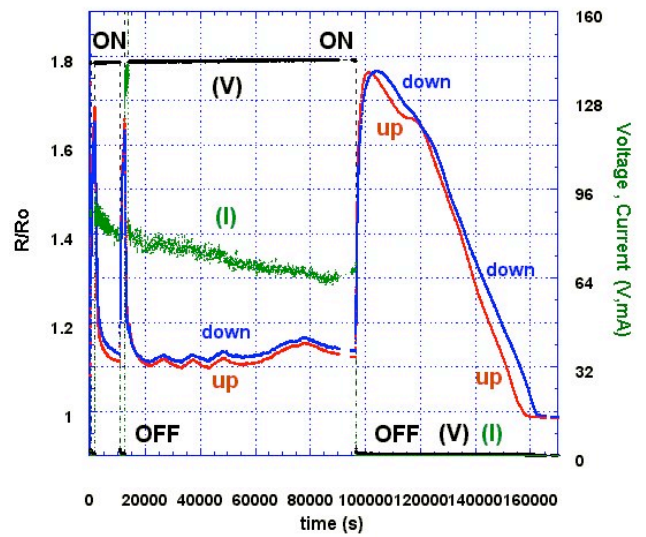


Fig. 6 – Test OFF/ON with Sr (+ very low Hg) and de-loading run in OFF (Tab.1,4).

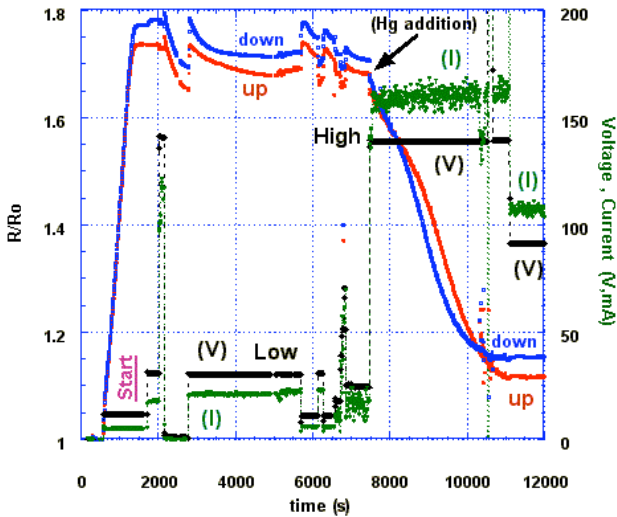


Fig. 7 – Test with Sr and Hg addition during the run with a L/H operation (Tab.1,7).

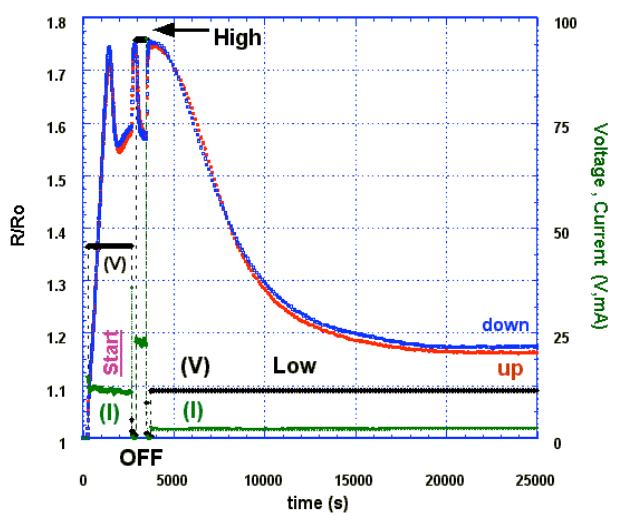


Fig. 8 – Test with Sr+Hg in an Ethylic solution; Start-OFF/H/L procedure (Tab.1,11).

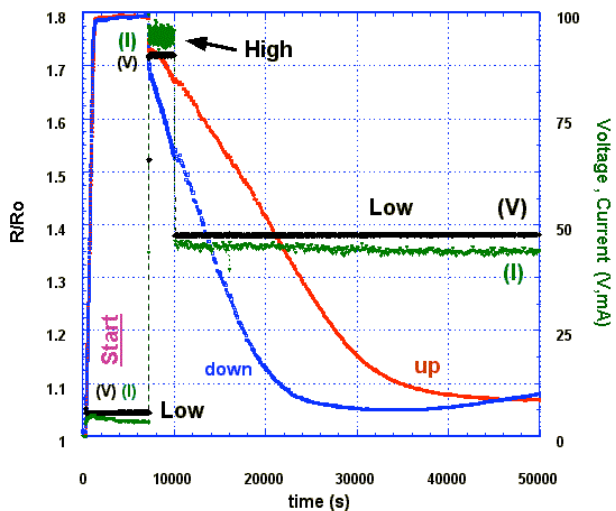


Fig. 9 – Test with Sr + Hg ($0.5\mu\text{M}$); Start-L/H/M procedure (Tab.1,15).

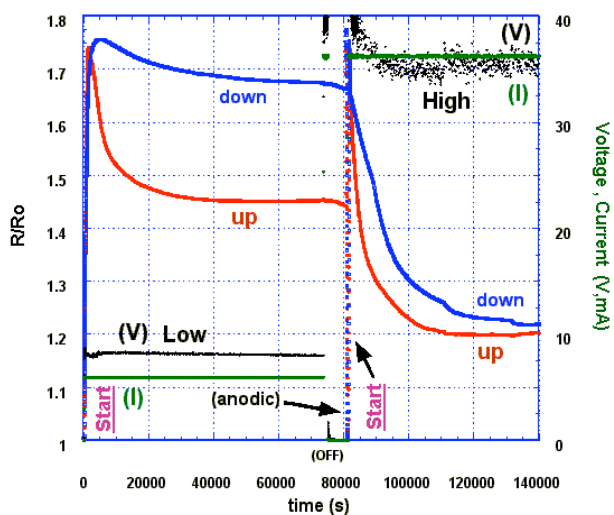


Fig. 10 – Test with Li+Hg ($1\mu\text{M}$); Start-L and Start-H procedures comparison (Tab.1,20).

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