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Swedish Radiation Safety Authority

Author: Richard Becker  
Hans-Peter Hermansson

Research

2011:34

Evolution of hydrogen by  
copper in ultrapure water  
without dissolved oxygen



## **SSM perspective**

### **Background**

The KBS-3 repository concept developed by SKB for disposal of spent nuclear fuel is based on a multi barrier principle for isolation of the fuel and to delay any escaping radionuclides. The concept is based on three barriers; copper canister, bentonite buffer and granitic bedrock. The copper canister will in this respect work as a corrosion barrier and completely isolate the spent nuclear fuel from the surroundings until failure of the 5 cm thick copper canister by corrosion or mechanical loads occurs. Copper as canister material was chosen by SKB 1978 in the KBS-2 report based partly on that copper was stated to be thermodynamically immune in water without dissolved oxygen (anoxic water). Thermodynamic immunity of copper in anoxic water implies that corrosion of copper only will take place if species like sulphide which cause copper corrosion are present in anoxic groundwater.

The immunity of copper in anoxic pure water has since the end of 1980's been discussed in Sweden based on original experimental work by Hultqvist and co-workers, who provided evidence of hydrogen gas evolution when copper was exposed to anoxic pure water. Evolution of hydrogen gas during exposure of a metal in an aqueous solution is usually clear evidence that corrosion of the metal occurs. The difficulty in obtaining unequivocal evidence is in this case of copper that hydrogen gas will be formed at a maximum partial pressure significantly lower than 1 bar. Bubbles of hydrogen gas will therefore not be formed during exposure. In order to detect hydrogen gas evolution when copper is exposed to pure anoxic water, Hultqvist and coworkers used a specially designed experimental set-up.

Research results presented in this report is one part of a larger project jointly financed by SSM and the Finnish research program on Nuclear Waste Management, KYT.

### **Objectives**

The objective with this research project was to increase knowledge in the area of copper corrosion in pure anoxic water in order to facilitate a science based review of the KBS-3 concept.

### **Results**

One result from this research was that the experimental difficulties of repeating Hultqvist and co-workers work are considerable and were initially underestimated. The main challenge was to obtain satisfactory tightness of all connectors involved in the experimental set-up. The problems with leakage resulted in that the research program originally planned for in this project had to be reduced considerable.

Nevertheless, experimental work conducted in this project has in principle reproduced parts of Hultqvist and co-workers work. Exposure of copper in pure anoxic water resulted in a measureable gas production

rate. Confirmation of hydrogen gas formation was achieved by mass spectrometry analysis. A reference test by exposing a platinum foil in anoxic pure water with the same surface area as for the copper was conducted. The exposure of platinum foil did not cause any evidence of gas evolution. This shows that the kind of immersed metal rather than a metal surface itself is important in order for gas to be evolved. The most obvious explanation for the results in this work is consequently that corrosion of copper occurs in pure anoxic water. Recently published thermodynamic calculations by Macdonald also suggest that copper cannot be considered as thermodynamically immune in the presence of pure anoxic water.

It must be emphasized that even if copper will corrode in pure anoxic water, firm conclusions about how copper canisters at repository depth will behave with regard to corrosion cannot be drawn. The rate of corrosion will be affected by the thermodynamic driving force associated with hydrogen gas formation but this has not been addressed in this study. Corrosion of copper canisters will be determined by a very complex interaction between copper, buffer material, geochemical and groundwater flow condition at repository depth.

#### **Need for further research**

The confirmed evolution of hydrogen gas when copper is exposed to anoxic pure water shows that more work is needed to explain the mechanism for gas evolution. Furthermore, this also shows that the process understanding how copper will behave chemically in a repository environment need to be elucidated further. In this respect more experimental and theoretical work is needed in environments which more closely represents the conditions found in the planned repository.

#### **Project information**

Contact person SSM: Jan Linder

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Strålsäkerhets  
myndigheten

Swedish Radiation Safety Authority

**Authors:** Richard Becker and Hans-Peter Hermansson  
Studsvik Nuclear AB, Sweden

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This report concerns a study which has been conducted for the Swedish Radiation Safety Authority, SSM. The conclusions and viewpoints presented in the report are those of the author/authors and do not necessarily coincide with those of the SSM.

# Summary

The thermodynamic immunity of copper in ultrapure water without dissolved oxygen has been under review since it was suggested that copper, in contradiction to classic thermodynamics, does in fact corrode in this environment. Experiments by Swedish researchers have shown that hydrogen seems to be generated during exposure of copper in ultrapure water without dissolved oxygen. To explain the results it has been proposed that the thermodynamic stability of copper is influenced by a previously unknown and uncharacterized metastable copper(I) hydroxide phase. The existence of this phase would make corrosion of copper in ultrapure water without dissolved oxygen plausible. However, previous attempts to verify these results using different methods have so far not been successful.

Experiments have now been performed at Studsvik to further study if hydrogen gas can be evolved via a reaction of copper and ultrapure water without dissolved oxygen. The experiments have been performed in a two chamber system separated by a thin palladium foil which allows for hydrogen to pass through it. Copper and water is placed in one of the chambers and the other is evacuated. A pressure increase on the other side of the palladium membrane is attributed to corrosion of copper. The same techniques were used by the Swedish researchers mentioned above.

Three experiments were performed. In two of the tests copper was exposed in ultrapure water without dissolved oxygen and in the third platinum was used as a reference experiment without the possibility for hydrogen evolution. The results obtained showed that hydrogen gas was evolved during the exposure of copper in ultrapure water without dissolved oxygen, but not during the reference experiment with platinum exposed in the same experimental setup and under the same experimental conditions.

The total amount of hydrogen detected after the test is lower than what would be possible with respect to the copper amounts found in the test solutions after the experiments. The same observation was made in previous experiments mentioned above. In addition in this work the test solutions were shown to contain unexpected amounts of metals. However, the gas and the test solution analyses and their interpretations are complicated and were originally outside the scope of this work.

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# Introduction

The Swedish strategy for the disposal and final storage of spent nuclear fuel is based upon the KBS-3 plan, which has been developed by the Swedish Nuclear Fuel and Waste Management Company (SKB), see e.g. SKB (1983). In 2006, SKB presented the SR-Can technical report (SKB 2006) which summarized the long-term safety effect of the KBS-3 plan. The conclusions from this report are that the three barriers of the KBS-3 plan together effectively will avoid any release of nuclides during the life-time of the repository. The three distinct barriers of the KBS-3 concept that will isolate and contain the waste are:

- a copper canister,
- a bentonite clay buffer, and
- the crystalline bedrock.

The first barrier is the canister that will enclose the spent fuel, and this barrier is presumed to be the most important barrier to prevent leakage of radioactive nuclides from the nuclear fuel to the biosphere. The canister comprises a 5 cm thick outer shell made up of phosphorus alloyed oxygen free high conductivity (OFHC) copper and an inner load bearing part made of ductile cast iron. The outer copper layer is supposed to constitute the primary anti-corrosive layer of the canister. Copper was chosen due to the fact that it is assumed to be thermodynamically immune to corrosion when in contact with ultrapure water without dissolved oxygen. This immunity is based upon the fact that any oxidation process for copper involves a positive change in the Gibbs free energy, where a negative change is required by the laws of thermodynamics for a reaction to occur spontaneously. Additionally, large metallic copper deposits have been found throughout various locations in the world, which also supports the view that metallic copper is stable with respect to the repository reference environment. This puts copper in the same group as the noble metals such as gold, compared to other more ignoble metals that only ever have been found in nature as oxides/hydroxides and/or sulphides. To a large extent, the KBS-3 concept is said to fulfil all requirements set forth by the regulatory agency in Sweden for the storage of spent fuel both for the main reference environment as well as for any transient environments that could occur over the expected 100 000 years of lasting storage integrity.

However, there are a few areas where more research is needed to verify the solidity of the KBS-3 plan, and one such area is the integrity of the copper canisters. The question of the thermodynamic immunity of copper in pure water was initiated by Swedish scientists in the late 1980ies (Hultquist 1986) and then debated over period of time, see e.g. Simpson *et al.* (1987), Hultquist *et al.* (1989) and Eriksson *et al.* (1989). The findings reported by Hultquist in 1986 were independently investigated and the experiment repeated by Möller (1995), but his experiment did not show any evidence of oxidation of copper in ultrapure water without dissolved oxygen. Möllers conclusions were that only a negligible corrosion of copper had occurred, and this corrosion was due to the presence of oxygen at the initiation of the experiment.

The question concerning the thermodynamic stability of copper in ultrapure water without dissolved oxygen was more or less dormant until elevated again in 2007 (Szakálos *et al.* 2007). This report has generated some attention and concern from the media due to the important role of copper in the KBS-3 plan concerning the disposal of spent fuel. The authors propose that copper actually does corrode in ultrapure water without dissolved oxygen under the evolution of hydrogen gas, and they hypothesize on numerous possible hypotheses concerning the oxidation process of copper. One suggestion involves a new, previously unknown and so far thermodynamically uncharacterized, copper(I) hydroxide phase. The results from the paper by Szakálos *et al.* in 2007 has been summarized and discussed in a previous SSM report (Apted *et al.* 2009). Szakálos *et al.*

has since their paper in 2007 published additional papers on the subject of corrosion of copper in ultrapure water without dissolved oxygen (see e.g. Hultquist *et al.* 2009, 2011).

The experiments performed by Szakálos *et al.* include amongst other a pressure gauge experiment, where gas is evolved when ultrapure water without dissolved oxygen is in contact with a sheet of copper foil. This gas passes through a palladium membrane, and accumulates in an evacuated chamber where the pressure is monitored. Hydrogen easily penetrates through the palladium membrane, so an increase in pressure above the palladium membrane is interpreted as accumulation of hydrogen gas. The gas that has accumulated has been verified by Szakálos *et al.* to be hydrogen using mass spectroscopy analysis. The statement of Szakálos *et al.* is that the hydrogen is produced as a product when copper corrodes while in contact with ultrapure water without dissolved oxygen. All their published data, according to their conclusions, seem to support their findings that copper does corrode in ultrapure water without dissolved oxygen under the evolution of hydrogen.

## Aim of this project

The aim of this project has been to repeat the pressure gauge experiment that originally was published by Szakálos *et al.* (2007) and to either verify or disprove the results that were obtained by them concerning the corrosion of copper in ultrapure water without dissolved oxygen. The work has been part of a collaboration between Studsvik Nuclear AB, the Swedish Radiation Safety Authority (SSM), Aalto University in Helsinki, Finland and VTT Technical Research Centre of Finland.

The work was divided into separate tasks:

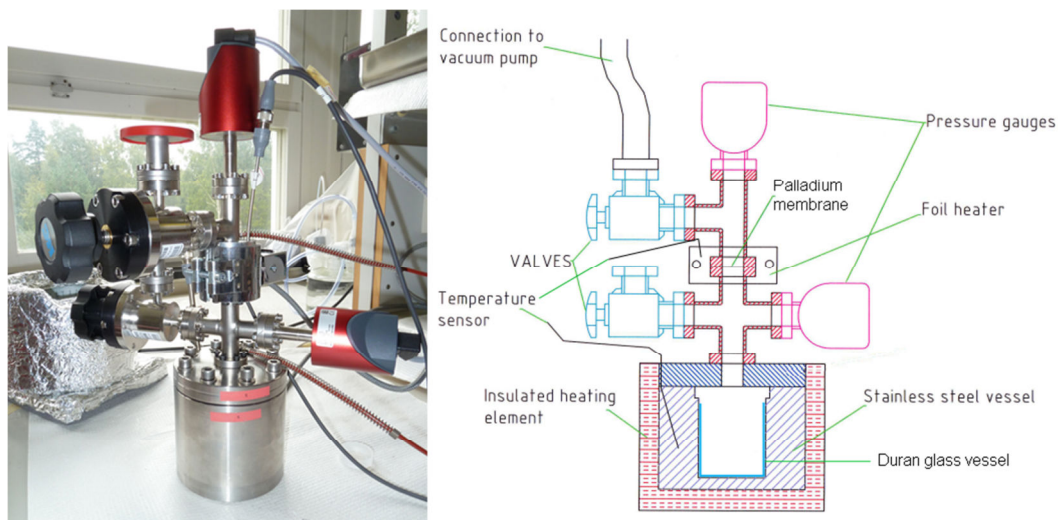
1. Preparation and testing of the experimental equipment including the test vessel, which should be a similar equipment set-up to that which was used by Szakálos *et al.* (2007).
2. Experimental part at Studsvik Nuclear:
  - a. Two exposures of copper in ultrapure water without dissolved oxygen with pressure monitoring according to the same set-up as Szakálos *et al.* (2007).
  - b. A reference experiment performed in the same manner as for the copper experiments but where the copper foil is replaced by a platinum foil.
3. Evaluation and conclusions.

# Experimental

## Equipment set-up

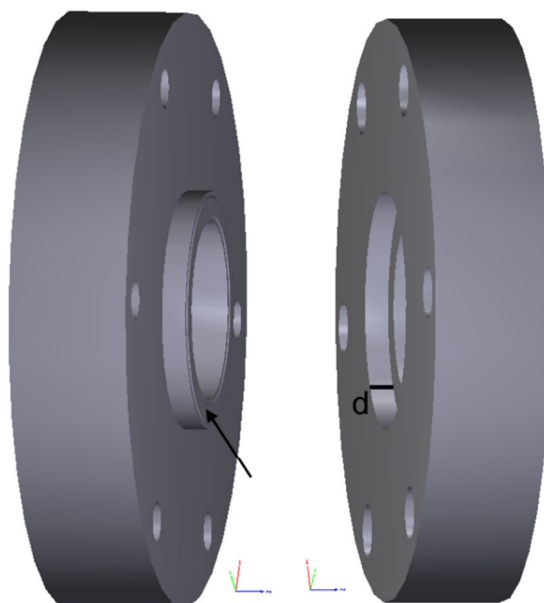
The experimental equipment was manufactured by Aalto University, Finland, while the final assembly and testing was performed by Studsvik Nuclear.

Two ultrahigh vacuum chambers connected via a palladium membrane were used in this experiment. The experimental set-up with these two chambers is shown in Figure 1.



*Figure 1* Left image shows the experimental equipment used in the present corrosion tests. Right image (courtesy of Seppo Jääskeläinen, Aalto University, Finland) shows a schematic drawing of the same equipment.

The set-up consists of a lower (larger) chamber with a Duran glass vessel inside the chamber. A four way-tube is connected to this chamber which in turn is connected to a valve, a pressure sensor and the palladium membrane. The total volume of the chamber and the four-way piece below the palladium membrane is roughly  $190 \text{ cm}^3$ . The palladium membrane is located inside a special holder that was manufactured to form a tight seal. This holder has no gaskets but instead uses the geometry of the holder and the raised edges to form a tight seal to the membrane, see Figure 2.



*Figure 2* The two parts of the palladium holder (image courtesy of Seppo Jääskeläinen, Aalto University, Finland).

The palladium membrane is placed in between the two holder parts and the raised edge (indicated with an arrow in Figure 2) presses on the membrane and forms the tight seal. The integrity of the seal of the original part was checked after which the distance between the two parts (indicated with a  $d$  in Figure 2) was increased by  $5\ \mu\text{m}$ . The integrity of the seal was checked again and the distance was increased by a further  $5\ \mu\text{m}$ . This process was repeated until the seal was deemed adequate.

The surface of the palladium exposed inside the holder has a diameter of 16 mm. The holder is in turn attached to a three-way tube which is connected to a valve and a pressure sensor. The total volume above the palladium membrane is approximately  $18\ \text{cm}^3$ . For all flanges and connectors, copper gaskets were used, except for the previously mentioned special holder for the palladium membrane where no gaskets were used. Before assembly, all parts were baked in a furnace at  $110\text{--}130^\circ\text{C}$  for 48 hours and allowed to cool inside an exicator. After assembly, each chamber was continuously evacuated for at least seven days to evacuate any moisture absorbed by the parts.

The integrity of the whole experimental set-up was checked both by evacuating the chambers and monitoring the pressure, and using helium leakage equipment after the experimental set-up had been assembled. The monitoring of the pressure of the chamber above the membrane after evacuation revealed a very low pressure increase, in the order of  $0.0002\text{--}0.0003\ \text{mbar}/24\text{h}$  which was assumed to be low enough not to have an effect on the results. The helium leakage tests revealed no leaks in the system. However, after the He-leakage test, two seals had to be broken to insert the water and the copper foil to initiate the experiment: the lid connected to the bottom chamber and the four-way piece connected to the lid. To make sure that a good vacuum could be achieved after these parts were installed and the seals were restored using new copper gaskets, the tightness between each connection was checked both via the torque used to tighten the screws and by checking the distance between the two parts using a feeler gauge. The same torque and distance were then used when reassembling the equipment before each subsequent test was initiated, and the pressure was monitored for several days before the temperature was increased. The integrity of the lower chamber can thus not be guaranteed but the fact that the same torque and distances were used in subsequent experiments and the fact that no pressure increase was observed (although please note that the pressure meter used for the lower chamber has measurement range between  $1.1\ \text{bar}$  and  $10^{-1}\ \text{mbar}$ ) indicates that no leakage occurred in the lower chamber.

## Materials

All parts of the experimental equipment were manufactured in stainless steel (316L) with the exception of the heat transferring piece between the heating element for the palladium holder and the actual holder which was manufactured out of aluminium. This piece only functions as a heat-transferring element between the heater and the membrane holder and is never in contact with experimental environment.

The glass vessel inside the lower chamber was made of Duran glass.

All gaskets were made out of OFHC copper.

The palladium membrane was manufactured from 99.95 % 0.1 mm thick palladium foil (Temper: As rolled). It was polished using a 1200 mesh SiC grinding paper before inserted in the holder. A special fixture out of Teflon was constructed for this purpose. This allowed the membrane to be polished only on the surface which would be exposed in the holder and not on the membrane surface which forms the seal inside the special holder.

The copper foil used in the experiment was 0.1 mm thick 99.95+ % OFHC (Temper: Hard). It was polished using a 1000 mesh SiC grinding paper before exposure.

The platinum foil used in the reference experiment was 0.1 mm thick 99.99 %. It was polished using a 1000 mesh SiC grinding paper before exposure.

## Measuring equipment

The pressure in the set up was monitored:

- In the lower chamber using a PTR24602 active capacitive transmitter CMR 361 (measuring range 1100 mbar to  $10^{-1}$  mbar).
- In the upper chamber using a PTR24632 active capacitive transmitter CMR 364 (measuring range 1.1 mbar to  $10^{-4}$  mbar).

The temperature in the set up was monitored:

- In the lower chamber by a PT-100 sensor that was placed inside the chamber wall.
- In the palladium membrane by a PT-100 sensor placed inside the aluminium heat-transfer piece between the holder and the heating element.

The vacuum pump used to evacuate the chambers was a HighCube 80 Eco Turbo pumping station.

The gas mass spectrometer (MS) used is an InProcess Instruments GAM-400 equipped with a batch inlet system and an on-line inlet system. The instrument has a quadrupole mass filter.

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) was conducted by injecting the eluents into a Perkin Elmer Elan 6100 DRC II Inductively Coupled Plasma Mass Spectrometer (ICP-MS), installed in a glove box. The instrument is equipped with a Dynamic Reaction Cell (DRC), which is used for elimination/reduction of interferences. The DRC mode was used for the iron analyses of the samples.

# Experimental performance

A total of three experiments were conducted using the experimental set-up: two using a copper foil and one using a platinum foil (to serve as a reference).

For each experiment, approximately 140 cm<sup>2</sup> of the copper or platinum foil was placed inside the glass vessel in the lower chamber together with 90 cm<sup>3</sup> of ultrapure water. The chamber above the palladium membrane had been evacuated for several days to the lower limit of the pressure sensor (< 10<sup>-4</sup> mbar). After assembly and insertion of water and foil, the lower chamber was evacuated using an equalization technique where a secondary vessel (with a volume around ten times that of the chamber) was placed between the pump and the chamber. This additional vessel was evacuated for approximately five seconds after which the pressures between the chamber and the additional vessel were allowed to equalize for approximately 60 seconds. The connection between the two was then closed and the process was repeated for a total of ten times which brought the pressure of the lower chamber down to about 30 mbar, a pressure which is assumed to consist only of water vapour.

For all experiments, after the evacuation process of the lower chamber, the pressure was monitored in both chambers for about three days without any increase in temperature to acquire a stable pressure background level. When such a level had been confirmed, the temperature was raised by one of the following two methods.

## 1) Cu run-1

For the first copper experiment (run-1) the heater for the palladium foil was set to 63°C using a heating rate of 15°C/h, while the heater for the lower chamber was initiated approximately 30 minutes later and was set to 60°C using a heating rate of 10°C/h.

## 2) Cu run-2 and Pt reference-run

For the second copper experiment (run-2) as well as for the platinum experiment, the heater on the palladium foil was kept at 63°C throughout the entire process of preparation, *i.e.* during the initial evacuation of the lower chamber and the following three day background monitoring to acquire a stable background level. The heater of the lower chamber was then set to 60°C using a heating rate of 10°C/h.

The temperature of the membrane heater was kept a few degrees higher than that of the lower heater to avoid any condensation of water on the palladium membrane.

After the temperature in the lower chamber was raised, the set-up was left for approximately two weeks. The pressure of the lower chamber increased with this temperature increase due to the vapour pressure of the water. The final pressure of the lower chamber was around 160 mbar throughout all three experiments. During the entire experimental procedure, an automated logger kept track of the two temperatures, the two pressures and the ambient temperature in the room. When two weeks had passed, the upper chamber was evacuated for roughly 30 seconds. The set-up was then left for approximately one additional week.

After a total of three weeks of exposure, the heater for the lower chamber was turned off and allowed to cool to ambient temperature. For the two copper experiments, the gas which had accumulated above the palladium membrane was collected and checked qualitatively for the presence of hydrogen gas. The pressure above the palladium membrane after the platinum reference experiment was too low to allow for a gas mass spectrometry analysis.

Between each run, the upper chamber was continuously evacuated for several days to remove any hydrogen which might have accumulated inside the palladium membrane. The same membrane and the same glass vessel were used in all experiments. The glass vessel was rinsed thoroughly with acetone, ethanol and water between each experiment.

The water from the glass vessel in the lower chamber was collected and analysed using ICP-MS after each exposure together with an ultrapure reference water sample. No filtration of the samples occurred before the analyses. After the final exposure, concentrated nitric acid was used to rinse the glass vessel. This nitric acid was diluted and analysed using ICP-MS, and the same analyses were also performed on a blank diluted nitric acid sample for reference.

# Results

Three experiments have been performed to analyse if hydrogen gas is produced by copper in contact with ultrapure water without dissolved oxygen in accordance with Szakálos *et al.* (2007). The results are presented below divided into three sections: from pressure data, from the gas Mass Spectrometry (MS) analyses and from the Inductively Coupled Plasma Mass Spectrometry (ICP-MS).

## Results from the pressure gauge experiment

Three tests, two using individual copper foils and one using a platinum foil to function as a reference experiment, were conducted. The experimental parameters have been given earlier in the experimental section.

The first copper experiment (Cu run-1) was conducted according to the experimental procedure already given. The pressure in the chamber above the palladium membrane, the temperature in the heater surrounding the membrane and the temperature in the heater around the lower chamber were continuously monitored, see Figure 3 below.



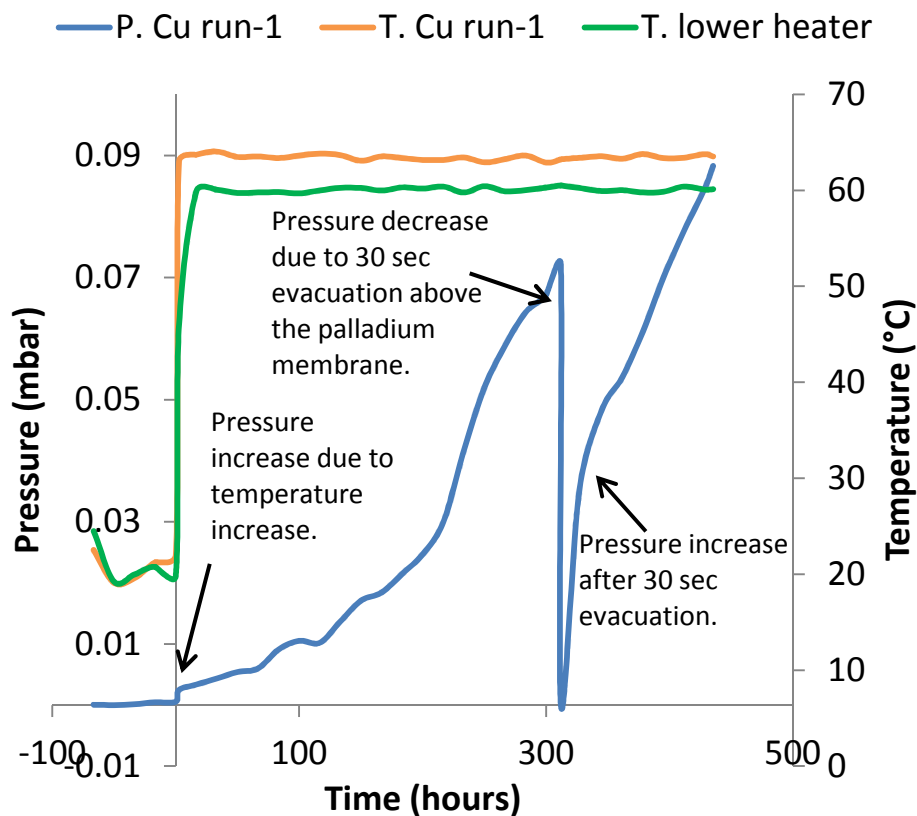


Figure 3 Pressure in the upper chamber together with temperatures in the lower heater and the heater around the palladium membrane for the first copper experiment.

The temperature of the heater around the membrane was initiated at time = 0 hours. A small, rapid, increase in pressure is observed due to this temperature increase, and this has been indicated by an arrow in Figure 3. This can be explained by the universal gas law, which states that an increase in temperature at constant volume should give an increase in pressure. After this initial increase, the pressure increased with an escalating rate until a steady-state occurs. The chamber above the palladium membrane was evacuated after approximately two weeks after which the pressure quickly increased again reaching the steady-state rate more quickly than at the initiation of the experiment.

Figure 4 below shows the pressure in the chamber above the palladium membrane, the temperature in the heater surrounding the membrane and the temperature in the heater around the lower chamber, for the second copper experiment (Cu run-2).

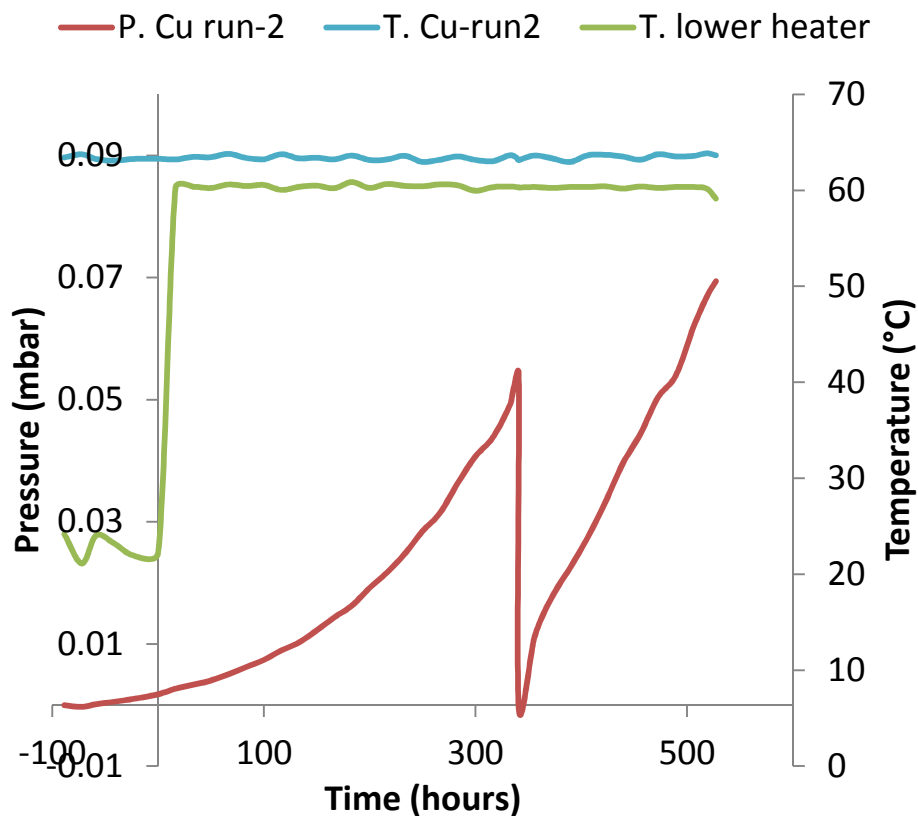


Figure 4 Pressure in the upper chamber together with temperatures in the lower heater and the heater around the palladium membrane for the second copper experiment.

The results from the second copper experiment are comparable to those obtained from the first copper experiment with similar behaviour in pressure increase rates.

Figure 5 below shows the pressure in the chamber above the palladium membrane, the temperature in the heater surrounding the membrane and the temperature in the heater around the lower chamber, for the last experiment, the platinum reference experiment. The observed pressure increase during the initial background monitoring of the equipment set-up has been added as a reference curve.

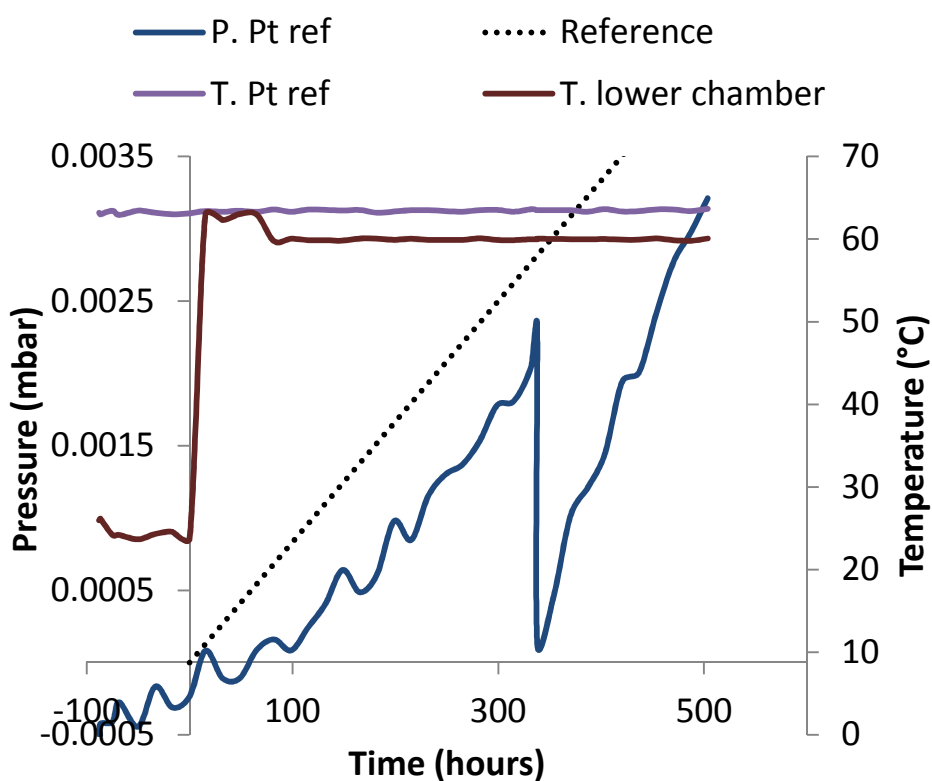


Figure 5 Pressure in the upper chamber together with temperatures in the lower heater and the heater around the palladium membrane for the platinum reference experiment. The negative value is a result from the measuring equipment which has a lower limit of  $10^{-4}$  mbar. A pressure below this is given as a negative value. Please note the change in scale on the y-axis for this graph compared to the previous graphs for the copper experiments.

The pressure above the palladium membrane did not increase above the calculated levels from the observed pressure increase during the background monitoring of the equipment when platinum was used instead of copper.

Figure 6 below shows the pressure in the upper chamber above the palladium membrane for all three experiments on the same y-axis.

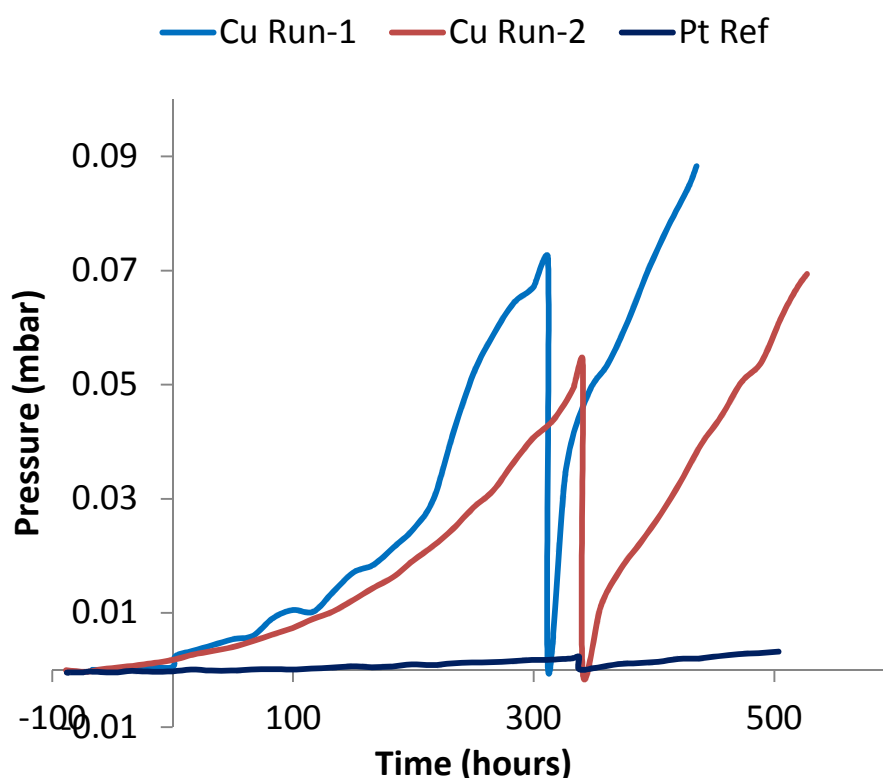


Figure 6 Pressure data for the upper chamber during Cu run-1, Cu run-2 and Pt-ref.

It is evident that there is a clear difference between the two experiments performed with copper compared to the reference experiment performed with platinum. Table 1 below shows the maximum pressure levels achieved just before the evacuations (after about two weeks), as well as at the end of the exposure (after about three weeks in total). The data in Table 1 indicates that the pressure has increased by a factor of 25-30 when using copper compared to the reference platinum experiment. The amount of hydrogen that would correspond to the listed pressure levels above the palladium membrane has been included in Table 1. These amounts were calculated to acquire possible amounts of hydrogen that could have been accumulated above the membrane under these experimental conditions. The calculations were performed using the universal gas law equation ( $p \cdot V = n \cdot R \cdot T$ ) assuming a temperature of 63°C and a volume of 18 cm<sup>3</sup> (the volume of the experimental equipment above the palladium membrane)

**Table 1** Pressure values reached just before the evacuation as well as at the end of the exposure for the two Cu experiments and the Pt reference experiment. The amount of hydrogen (moles total) was calculated from the pressure levels using the universal gas law equation.

	Time (h)	P <sub>just before evac.</sub> (mbar)	H <sub>2</sub> (mole)	Time (h)	P <sub>end of exp.</sub> (mbar)	H <sub>2</sub> (mole)
Cu run-1	~312	0.072	4.6*10 <sup>-8</sup>	~435	0.088	5.7*10 <sup>-8</sup>
Cu run-2	~340	0.054	3.5*10 <sup>-8</sup>	~526	0.068	4.4*10 <sup>-8</sup>
Pt-ref	~338	0.002	-	~500	0.003	-

# Mass spectrometry results

Mass spectrometry was not originally a part of the experimental plan. Semi-quantitative gas mass spectrometry was performed on the gas in the upper chamber after the end of the full exposures for the Cu run-1 and Cu run-2 experiments. The analyses were semi-quantitative as the results would show a quantitative number for the level of hydrogen in the gas, but that *e.g.* the low amount of gas present would make any result more indicative of the presence of the gas than absolute values.

For the first Cu experiment, a sample flask was attached to the upper chamber via a T-connection. The flask and the connection were evacuated and the pressure was equalized between the chamber and the flask. For the second Cu experiment, the experimental set-up was instead, after the completion of the exposure, moved to the mass spectrometer where the set-up was connected directly to the spectrometer. No mass spectrometry analysis was performed after the platinum reference experiment as the pressure achieved above the palladium membrane was too low to allow this.

For the Cu run-1, two analyses were performed with a short time delay between the two. These results differ, as lighter elements typically get higher values early in an analysis while heavier elements get higher values after a time into the analysis. This is due to the fact that lighter elements easier penetrate into the mass spectrometer. Only one analysis was performed on the sample from the Cu run-2 experiment as it was attempted to accumulate more gas for the gas mass spectrometry analysis.

Table 2 below show the results from the mass spectrometry analyses on the gas in the upper chamber after the first (Cu run-1) and the second (Cu run-2) copper experiments. The helium in the results probably comes from the mass spectrometry equipment, which is flushed with helium between different analyses to remove any remaining gas before a new sample is introduced. Table 2 also gives the chemical composition of air for comparison. The levels of hydrogen after the Cu experiments are around 10 000 times higher than that of the amount of hydrogen in air, which clearly shows that hydrogen gas has accumulated above the palladium membrane in these experiments.

**Table 2** Mass spectrometry results (all data in atomic %). Water has been excluded from the results. The composition of air has been included for comparison.

	H <sub>2</sub>	He	N <sub>2</sub>	O <sub>2</sub>	Ar
Cu run-1 analysis 1	24.1 ±0.06	3.9 ±0.05	45.7 ±0.07	24.6 ±0.09	1.6 ±0.02
Cu run-1 analysis 2	12.3 ±0.18	4.4 ±0.10	53.4 ±0.29	28.0 ±0.27	1.9 ±0.07
Cu run-2 analysis 1	1.28 ±0.05	0	71.4 ±0.11	25.6 ±0.15	1.8 ±0.04
Air	0.00006	0.0005	78.1	21.0	0.93

# ICP-MS results

Chemical composition analyses using ICP-MS were performed on four different samples:

- A blank reference sample of the ultrapure water without dissolved oxygen before it had been used in any experiment.
- The water used in the Cu run-1 experiment.
- The water used in the Cu run-2 experiment.
- The water used in the Pt reference experiment.

Figures 7 and 8 below show the result from the ICP-MS analyses.

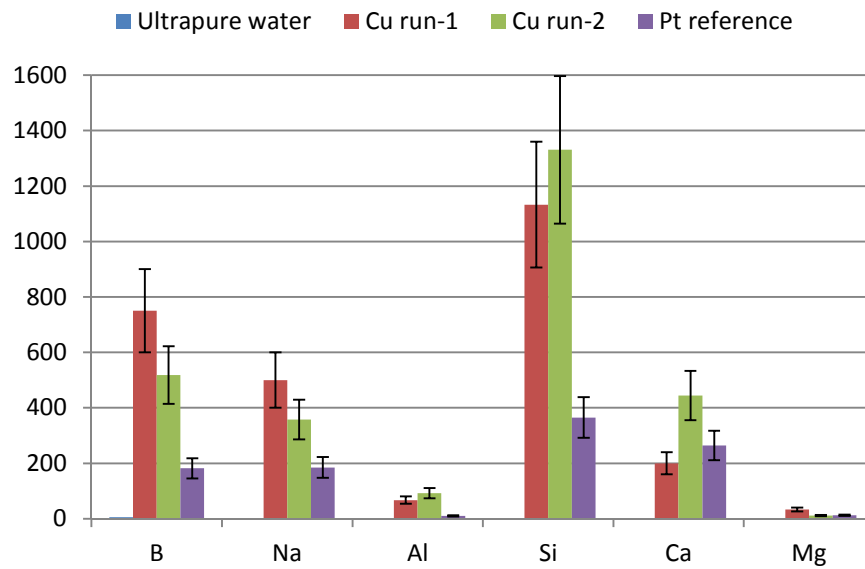


Figure 7 ICP-MS data (ppb) for the assumed glass elements. The levels for the ultrapure water analysis are too low to be visible in the graph (all were below 1 ppb, except B which was 6 ppb).

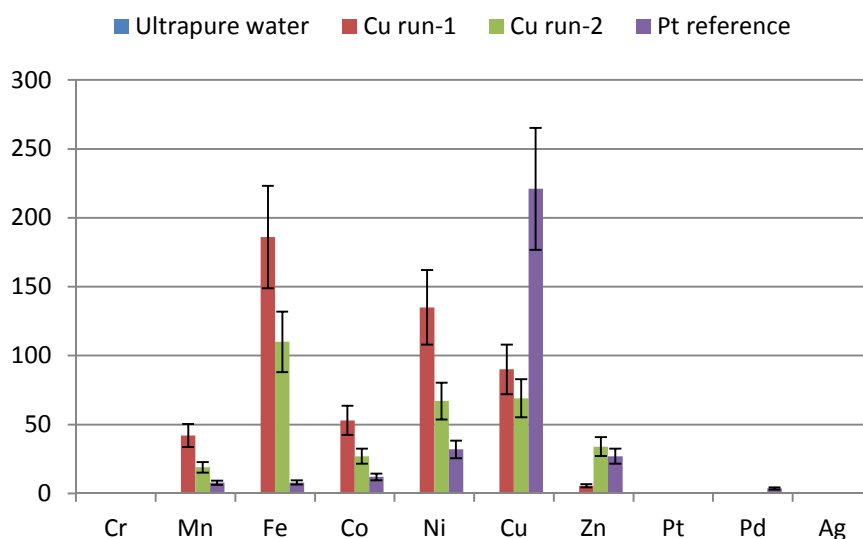


Figure 8 ICP-MS data (ppb) for the metallic elements. The levels for the ultrapure water are too low to be visible in the graph (all were below 1 ppb).

Elements typically found in glass (*e.g.* Si, Ni and B) were found in the water, especially following the Cu experiments, see Figure 7. These elements most likely originate from the glass vessel and have desorbed into the water during the exposure. If elements get desorbed from the glass vessel into the water, then it is considered possible for other elements to enter the glass. Metallic elements were found in the water following all analyses except the blank reference analysis, see Figure 8. Especially the presence of copper in the water after the reference platinum experiment was not expected, so additional analyses were performed to check if any metals indeed had been adsorbed by the glass vessel during the experiment. If this was the case, then these metallic elements could have been desorbed into the water during the reference Pt experiment.

The inside of the glass vessel was rinsed with nitric acid which was diluted and analysed. To acquire reference data for the analysis, the outside of the same glass vessel was also rinsed in the same manner. Additionally, the inside of a similar but unused glass vessel purchased at the same time as the one used in this experiments was rinsed in the same way. The results from these analyses are presented in Figures 9 and 10. They show the concentrations measured in the nitric acid after it was used to rinse the glass vessel. As it is impossible to know how large part of the adsorbed metallic elements in the vessel that has been leached or extracted by the nitric acid, it is not possible to transfer the measured concentrations in the nitric acid into concentrations in the vessel. The data sets are therefore comparable to each other, but not directly to the previous data concerning the concentration in the vessel water after exposure.

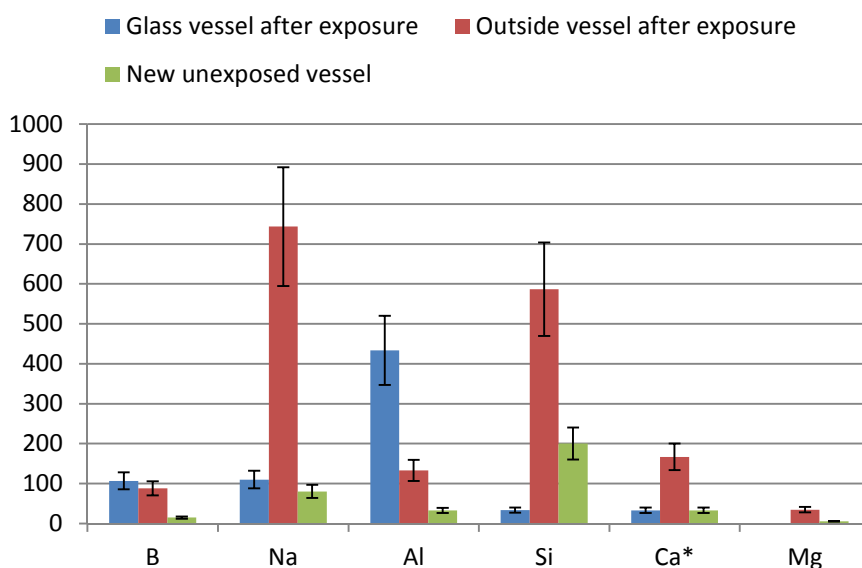


Figure 9 ICP-MS data (ppb) for the glass vessel for the glass elements.  
 \* Note that the values for Ca for two values are given as < 35 ppb due to dilution. The bar graphs are drawn at 35 ppb even though that the actual level probably is lower.

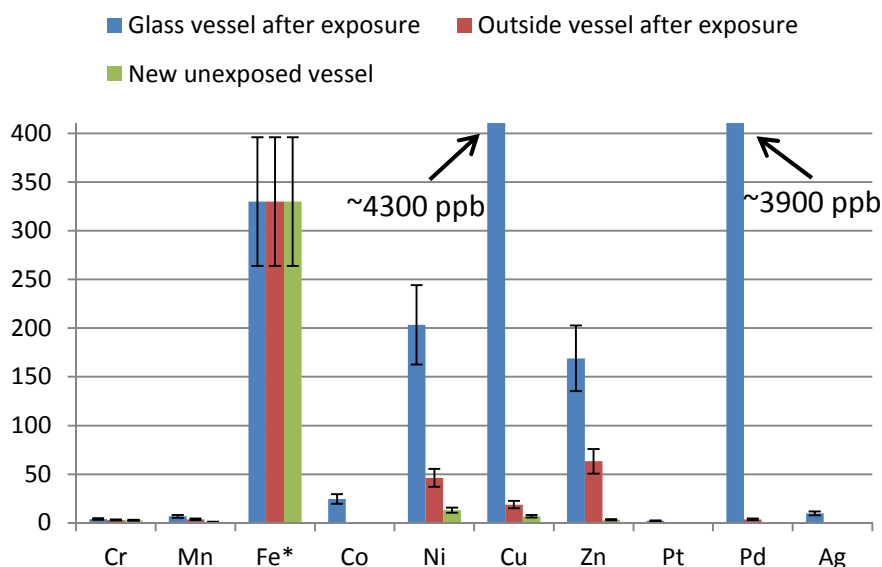


Figure 10 ICP-MS data (ppb) for the glass vessel for the remaining elements.  
 \* Note that the values for Fe are given as < 333 ppb due to dilution. The bar graphs are drawn at 333 ppb even though that the actual level probably is lower.

The analyses shown in Figure 9 show that some of the glass constituents seem to dissolve in the nitric acid. The amounts analyzed are all below 1 ppm. Figure 10 shows that considerably higher amounts of metals (Ni, Cu, Zn and Pd) were analyzed in acid used for rinsing the inner surface of the test vessel used for the exposures.

The copper foil used in the tests was analysed using ICP-MS to check its purity, see Table 3. Both a polished and a non-polished sample were analysed by dissolving a ~100 mg sample in nitric acid. No differences were observed for the two samples.



**Table 3** Measured levels of impurities in the copper foil (ppb).

	Level
Mg	< 1 000
Cr	< 100
Mn	< 100
Fe	< 1 000
Co	150
Ni	5 800
Zn	< 100
Pt	< 100
Pd	200
Ag	8 500

# Discussion

## Pressure gauge experiments

The pressure data clearly reveals a distinct difference in the pressure levels obtained above the palladium membrane when comparing the copper experiments with the reference platinum experiment, see Figure 6. These results are in agreement with the experimental results published by Szakálos *et al.* (2007) and suggest that hydrogen gas is produced when copper is exposed in ultrapure water without dissolved oxygen under the operational conditions in the experimental setup.

During the first copper experiment (Figure 3) the temperature was increased in the heater around the palladium membrane slightly before the temperature was increased in the lower chamber. This was so that the temperature always should be higher for the membrane so that no condensation should occur there. The pressure increased significantly after this to reach a value of approximately 0.072 mbar just before the chamber above the palladium membrane was evacuated. The pressure increase corresponds to about  $5 \cdot 10^{-8}$  mole of  $H_2$  gas (see Table 1) assuming that the entire gas volume has a temperature of  $63^\circ C$ , that is the same temperature as of the heater around the membrane.

A slower pressure increase is observed initially in the exposure. This is probably due to either that the palladium membrane needs to be fully saturated with hydrogen before a steady-state release rate is obtained to the upper chamber and/or a lower diffusion of hydrogen into the membrane as the hydrogen pressure builds up. The fully saturated palladium membrane used in this experiment can hold about  $1.6 \cdot 10^{-3}$  mole H (corresponding to  $0.8 \cdot 10^{-3}$  mole  $H_2$ ) assuming a 70 % saturation of the palladium membrane.

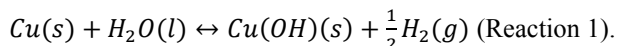
The pressure increase when steady-state has occurred (almost linear part in the curve) is calculated to be around 0.01043 mbar/24h. This pressure increase rate can be recalculated into moles of hydrogen gas produced per day using the universal gas law equation assuming a temperature of  $63^\circ C$  and a volume of  $18 \text{ cm}^3$  (the total volume above the palladium membrane) which then corresponds to  $6.7 \cdot 10^{-9}$  mole  $H_2/24h$ .

The experiment was continued after first the evacuation and the experiment was terminated after approximately three weeks. The pressure at termination was about 0.088 mbar. A quicker initial pressure increase after the first evacuation was observed compared to the start of the test. This has been attributed to hydrogen gas absorbed by the palladium membrane which is released as the pressure above the membrane is decreased. If this initial release is disregarded, the steady-state pressure increase (linear part of the curve) after the evacuation is estimated to 0.01086 mbar/24h which corresponds to  $7.0 \cdot 10^{-9}$  mole  $H_2/24h$ . The pressure increase rates reached at steady-state before and after the evacuation are in good agreement, which indicates that the evacuation process only momentarily disturbs the progression.

During the second copper experiment (Figure 4), the temperature in the heater around the palladium membrane was kept at  $63^\circ C$  throughout the preparation and background monitoring of the experiment. This was done to eliminate the effect of the initial pressure increase above the membrane as the temperature was increased. The result from the second copper experiment was similar to the first, although the resulting absolute pressure levels were somewhat lower (0.054 and 0.068 mbar respectively before and after the evacuation) compared to the levels obtained during the first copper experiment. If the same assumptions are made concerning the lower rate of pressure increase initially in the experiment, and the quicker pressure increase rate immediately after the evacuation, the pressure rates at steady-state can be calculated to 0.00704 and 0.00807 mbar/24h respectively before and after the evacuation. This corresponds to  $4.5 \cdot 10^{-9}$  and  $5.2 \cdot 10^{-9}$

mole H<sub>2</sub>/24h. These values are also in good agreement with each other although somewhat lower compared to the values obtained in the first copper experiment.

The reaction that produces during the copper exposure has been suggested (Hultquist 2011) to be oxidation of copper into copper(I) hydroxide according to reaction 1:



If several additional assumptions are made, e.g. that reaction 1 is the only source of hydrogen, that all hydrogen passes through the palladium membrane, that the flux of hydrogen gas through the palladium membrane is constant once steady-state has been achieved and that the corrosion process is not inhibited by any corrosion products, then the calculated rates of production of hydrogen could be transferred into possible rates of how fast copper is consumed, *i.e.* corrosion rates. These calculations are done using the density of copper (8.92 g/cm<sup>3</sup>) and the copper surface area used in the experiment (140 cm<sup>2</sup>). The results are presented in Table 4.

**Table 4** Rough estimates of copper corrosion rates from the hydrogen evolution rates.

	Est. production rate of H <sub>2</sub> (mole/24h)	Est. copper corrosion rate (mm/year)
Cu run-1 before evacuation	6.7•10 <sup>-9</sup>	2.5•10 <sup>-6</sup>
Cu run-1 after evacuation	7.0•10 <sup>-9</sup>	2.6•10 <sup>-6</sup>
Cu run-2 before evacuation	4.5•10 <sup>-9</sup>	1.7•10 <sup>-6</sup>
Cu run-2 after evacuation	5.2•10 <sup>-9</sup>	1.9•10 <sup>-6</sup>

It should be emphasized that the calculated corrosion rates are speculative as they are based on several assumptions, assumptions that has excluded several key rate-determining steps in the accumulation rate of hydrogen gas above the palladium membrane. They are aimed to be comparative to each other and roughly illustrate the small amounts measured in the test set-up. They should in no way be seen as reliable values for repository conditions, nor was the objective of this work to obtain such values.

The quick and easy penetration of hydrogen into *e.g.* the palladium membrane or the copper foil suggests that only part of the hydrogen evolved during the experiment will accumulate above the palladium membrane. It is however impossible to foretell how much will accumulate above the membrane and how much that will *e.g.* stay absorbed inside the palladium membrane or in the chamber below the membrane. Suggestions have been made that about 5-10 % of the evolved hydrogen gas passes through the palladium membrane and is detectable through the pressure increase, but these numbers have not been confirmed. Such factors have not been taken into account when the corrosion rates have been estimated.

For the reference experiment, platinum was chosen as a reference metal due to the noble properties of that metal: no corrosion should occur when platinum is placed in contact with ultrapure water without dissolved oxygen. The experiment was conducted in the same manner as for the second copper experiment, *i.e.* the temperature for the palladium membrane heater was kept at 63°C throughout the preoperational work. However, a small initial problem with isolation meant that the lower chamber had a slightly higher temperature than intended during the first few hours of the experiment. The temperature in the lower heater never exceeded the temperature in the heater around the palladium membrane which means that this should have a negligible effect on the experiment.

The pressure increase in this experiment (Figure 5) was observed in the order of 0.002 mbar for the first two weeks of exposure. This is in the same order as what was observed for the experimental setup during the initial background monitoring, and it suggests that no hydrogen gas has been evolved during the exposure of the platinum foil.

The overall results obtained in the present work clearly show a pressure increase in the chamber above the palladium membrane is obtained when copper placed in ultrapure water without dissolved oxygen in the experimental setup. The same pressure increase does not occur when the copper foil is replaced with platinum. The lack of pressure increase in the reference experiment indicates that the stainless steel vessel plays no role in the experiment, which has been suggested previously (Johansson 2008).

## Gas analysis

The results from the pressure gauge experiments showed that there is a clear difference between the experiments using copper and platinum, and that only copper causes a pressure increase above the palladium membrane. Semi-quantitative gas mass spectrometry was therefore performed to verify that it indeed was hydrogen gas which had accumulated above the palladium membrane, see Table 2.

The gas mass spectrometry showed that the levels of hydrogen in the analysed gas are several magnitudes larger than the levels of hydrogen which is present in air. Thus the hydrogen gas cannot come from a leak in the experimental set-up to the atmosphere either during the experiment or during the mass spectrometry analysis. The analysis should be used as qualitatively only showing that hydrogen is present in the upper chamber.

The analyses should not be used in a quantitative way. The amount of gas that was available for analyses are several factors lower than the recommended levels for normal analysis. Consequently, the accuracy is limited. The gas mass spectrometry analyses of the samples revealed large levels of O<sub>2</sub> and N<sub>2</sub> in the gas together with some water. The low amount of hydrogen gas available is most likely the cause for these results.

## Water analysis

The initial ICP-MS analysis showed that the ultrapure water used in the experiment did not contain any noticeable amounts of any elements before it was used. However, after the copper experiments, sub-ppm levels of Cu, Mn, Fe, Co, Ni and Zn were found in the water. For almost all of these metallic elements, their levels in the water were higher after the first copper experiment compared to after the second copper experiment.

Similar levels of some metallic elements were observed also after the Pt reference experiment. To further investigate this, the glass vessel was rinsed using nitric acid which then was analysed using ICP-MS. The results showed that the nitric acid used to rinse the glass vessel after the exposure contained large amounts of Cu and Pd together with sub-ppm levels of Ni, Zn, Co and Ag and possibly Fe. It is reasonable to assume that these low metal levels have been absorbed by the glass as *e.g.* Si, Na and B has desorbed from the glass into the water. Two blank analyses of the glass vessel did not show the same levels of metallic elements although all analyses gave a higher result for the analysis on the outside of the used glass vessel compared to the inside of the new vessel.

Thus, the ICP-MS results showed that metallic elements have been released into the water during the two copper experiments. The elements seem to have successively been absorbed into the glass as glass elements have been released into the water, see Figure 7. As the same glass vessel was used in subsequent experiments, some of the metallic elements could then be released into the water from the glass until equilibrium was reached, and even more metallic elements could be released when the glass vessel instead was rinsed with acid. The presence of metallic elements

inside the glass vessel could also explain why the second copper experiment gave a lower absolute pressure and a lower calculated corrosion rate: as metallic atoms/ions are released into the water from the glass vessel, the equilibrium of corrosion is shifted so that less corrosion can occur resulting in a lower absolute pressure above the palladium membrane.

The impurities in the copper foil consists mainly of Ni and Ag, so the presence of these elements in the water after the exposure could be explained by a dissolution or dissolving process in the foil that occurs simultaneously as the oxidation of copper. This observation of impurities thus strengthens the view that copper corrodes in ultrapure water without dissolved oxygen. The other impurities in the water and in the glass matrix cannot be explained solely by the same process, as their levels are too low in the foil to give the levels obtained in the water. It is however important to recognize that the ICP-MS analysis does not separate atoms from ions, so it is impossible to identify if the observed elements was present in the water during the analyses as ions (*e.g.* as a result from oxidation) or as metals (*e.g.* as a result from desorption or dissolution). In theory, any evolved hydrogen gas could be a result from the oxidation of any of the impurities instead of copper. This is perhaps not a likely situation as the levels of copper in the water after the exposure is at least ten times higher than the level of any impurities except palladium. A possible, although unlikely, source for these elements could be *e.g.* smearing or transfer during the preparation of the Cu foil. There is also the possibility that the ICP-MS analyses could have been contaminated, *e.g.* zinc from anthropogenic origin, as the levels observed are in the sub-ppm scale.

It is however not realistic to firmly and quantitatively discuss any possible corrosion of copper and the process of hydrogen evolution in this case until the origin and role of the impurities have been identified.

The ICP-MS results can be used to make assumptions concerning the maximum amount of hydrogen gas that could be produced by the corrosion of copper. The level of copper that was observed after the exposure can be recalculated into pressure produced by hydrogen via reaction 1. This pressure can be compared with what actually was observed and these values give that about 5-6 % of the maximum levels of hydrogen gas that could have been produced has been detected above the palladium membrane. This is in good agreement with the suggested value for how much hydrogen gas that could pass through the palladium membrane. It should however be noted that these estimates are based upon the assumption that all copper corrosion products have been released from the copper foil, a notion which is unlikely as some copper oxides undoubtedly has adhered to the foil.

## Corrosion products of copper

It has been proposed (Korzavyi and Johansson 2010) that CuOH(s) could form as a metastable phase during the corrosion of copper in contradiction with classic thermodynamics, but the proposed new copper(I) hydroxide phase is currently uncharacterized thermodynamically. There exists thermodynamic data for CuOH(aq) but not for CuOH(s) (Puigdomenech & Taxén 2000). Korzhavyi & Johansson (2010) have recently performed *ab initio* electronic structure calculations for several different possible copper oxides, copper hydrides and copper hydroxide which amongst other resulted in theoretical thermodynamic data for CuOH(s). Additionally, they found that CuOH(s) is metastable and can form in aqueous medium but will most likely decompose into copper(I) oxide and water.

The thermodynamic values calculated by Korzhavyi and Johansson could be used to hypothetically estimate a Pourbaix diagram for copper including the metastable phase. This has been done by recalculating the Gibbs free energy values published by Korzhavyi and Johansson into an equilibrium constant for the formation of CuOH(s). These calculations show that the stability region of the plausible Cu(OH) (s) phase, based upon the data published by Korzhavyi and Johansson, would cover most of the Cu<sub>2</sub>O stability region but would also extend slightly

below the hydrogen line. This postulation would mean that copper would not be stable in water even under “normal” circumstances, a proposal which evidently is wrong. However, the calculations should not be seen as anything other than the authors play with numbers to see if  $\text{CuOH(s)}$  theoretically could exist and if so where it would be located, based upon the thermodynamic data published by Korzhavyi and Johansson. If these calculation could be validated, it should be noted that the area of existence of  $\text{Cu(OH)(s)}$  under the stated conditions covers the hydrogen line. The possible evolution of hydrogen from copper in ultrapure water without dissolved oxygen is thus not contradicted by the hypothetical calculations.

# Conclusions

The results obtained from the pressure gauge experiment and from the mass spectrometry analyses are in line with those reported by Hultquist and Szakálos in that a pressure increase resulting from accumulation of hydrogen has been detected when copper is placed in ultrapure water without dissolved oxygen. The results obtained showed that hydrogen gas was evolved during the exposure of copper in ultrapure oxygen free water, but not during the reference experiment with platinum exposed in the same experimental setup and under the same experimental conditions.

The results obtained from the chemical analyses of the water after the exposures revealed small amounts of impurities which could influence the process of hydrogen evolution in this system. The origin and role of these impurities should be investigated further before any definite conclusions are drawn from the results.

The following conclusions can be drawn from the experiments performed:

- When copper is placed in ultrapure water without dissolved oxygen, a reaction occurs which releases hydrogen gas. The same reaction does not occur when platinum is used instead of copper in the same experimental set-up.
- It has been shown that it is in fact hydrogen gas which is produced during the exposure of copper in water without dissolved oxygen.
- The total amount of hydrogen detected after the copper test is lower than what would be possible with respect to the copper amounts found in the test solutions after the experiments. The same observation was made in previous experiments mentioned above. In addition in this work the test solutions were shown to contain unexpected amounts of metals. However, the gas and the test solution analyses and their interpretations are complicated and were originally outside the scope of this work.

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**Strålsäkerhetsmyndigheten**  
**Swedish Radiation Safety Authority**

SE-171 16 Stockholm  
Solna strandväg 96

**Tel:** +46 8 799 40 00  
**Fax:** +46 8 799 40 10

**E-mail:** [registrator@ssm.se](mailto:registrator@ssm.se)  
**Web:** [stralsakerhetsmyndigheten.se](http://stralsakerhetsmyndigheten.se)