Post-test examination of copper electrodes in dh 5 of the Prototype Repository

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Information about the copper electrodes

- Position of electrodes: In the upper block (C4) in deposition hole 5 (DA3551G01); at 269, 270 and 271°
- Size of each electrode: Ø60 x 100 mm; nominal surface area 232 cm²
- Two cables from each electrode with black, yellow-green, and red insulation ending in the G-tunnel (HG0023A02)
- Heat to the copper canister from 2003-05-08; the electrodes installed a few days earlier
- Real-time SmartCET® corrosion monitoring performed in periods starting in 2004
- The corrosion potential of the three electrodes was measured on 2011-02-04 as soon as the concrete barrier to the outer section had been removed

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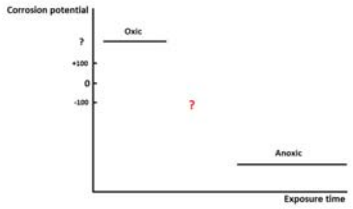
Oxic or anoxic conditions?

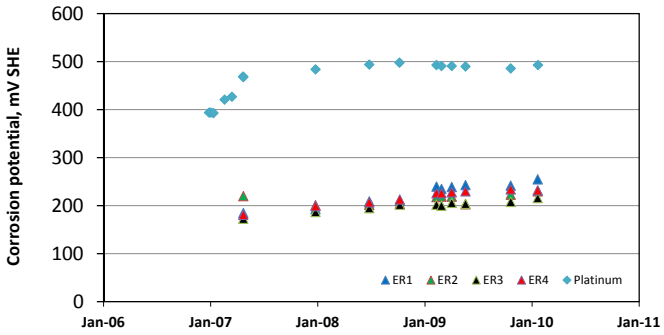
The exposure starts at oxic conditions, that is at a high redox potential and with high corrosion potentials of the order of 150 – 200 mV SHE.

After some time ? (see figure) anoxic conditions will be established with low redox and corrosion potentials. It is not obvious when this will occur.



3

Evolution of the corrosion potential with time for the ER sensors compared to the redox potential of the environment



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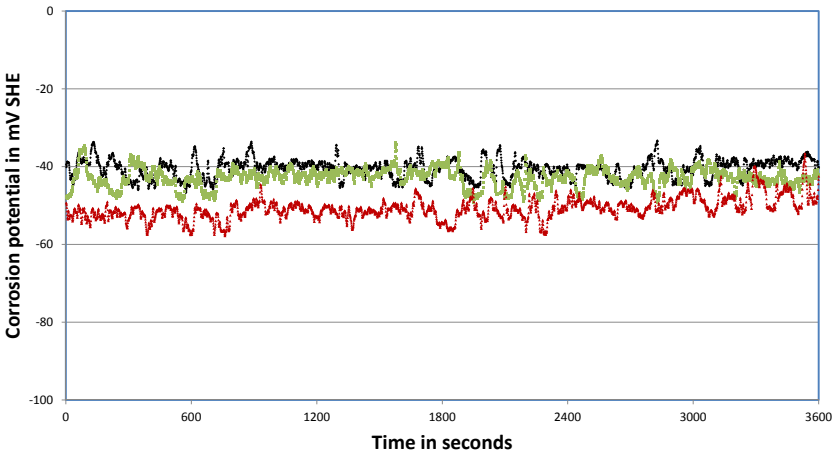
Äspö HRL – Prototype Repository – dh 5

Recorded corrosion potential of the copper electrodes on 2011-02-04



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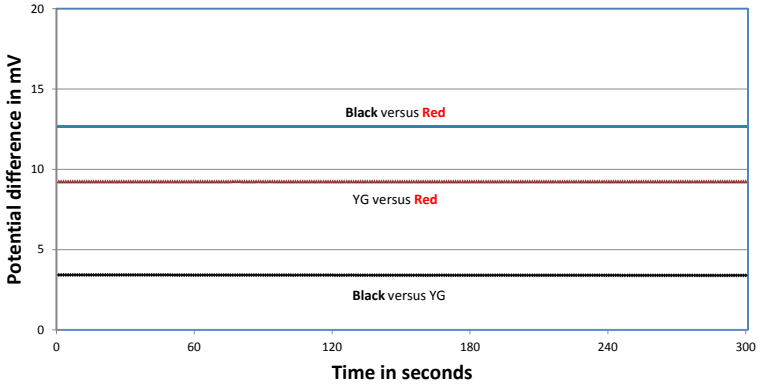
Potential differences between the copper electrodes in deposition hole 5 of the Prototype Repository



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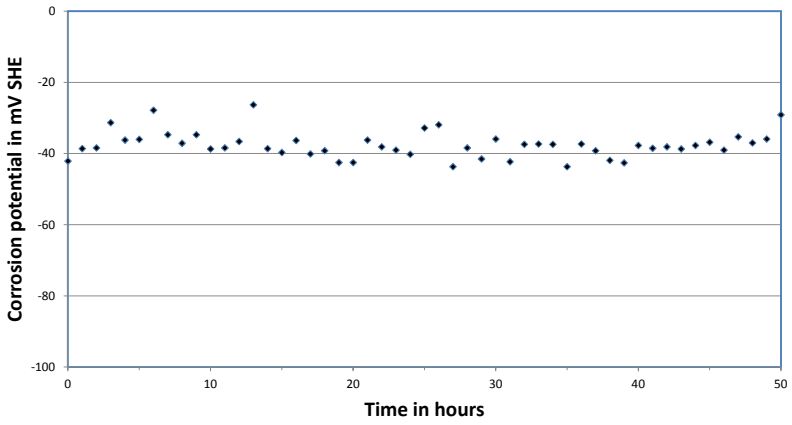
Corrosion potential of copper electrode "Black" in deposition hole 5 of the Prototype Repository



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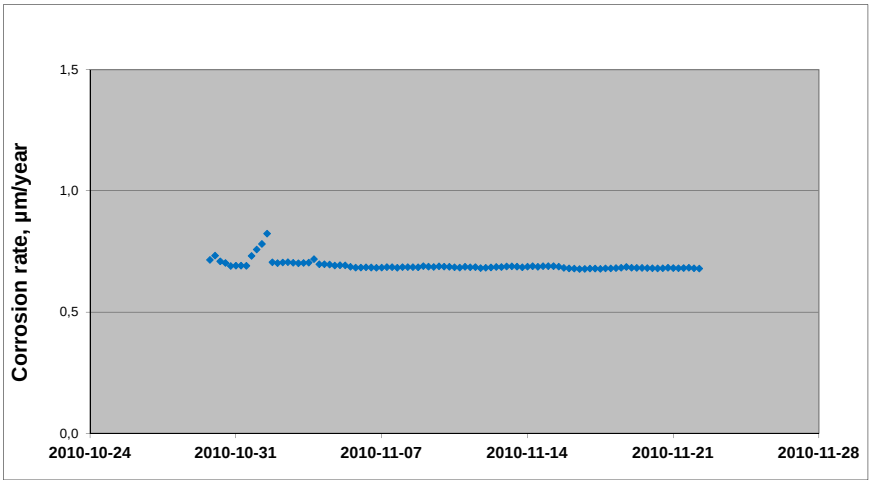
Äspö HRL – Prototype Repository – dh 5
Recorded corrosion rate on copper electrodes in 2010



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Information från driften av Prototypförvaret

Uppmätta korrosionshastigheter under första halvåret 2004 var lägre än 0,4 µm/år. Redan ett år senare uppmättes mer än dubbelt så höga värden och som högst 1,2 µm/år. Från 2005 har uppmätta korrosionshastigheter successivt avtagit ned till dagens nivå på 0,7 µm/år.

Under driften av Prototypförvaret har **dräneringen** av den inre och yttre sektionen temporärt **stängts av** en gång och problem med värmetillförseln till kopparkapslarna har föranlett avbrott i värmetillförseln och effektsänkningar. I början av **november 2004** stängdes dräneringen till den inre och yttre sektionen av och öppnades åter en månad senare. Omkring tre veckor efter det att dräneringen stängdes av noterades en påtaglig tryckhöjning i buffert och återfyllning från installerade total- och portrycksgivare. Värmetillförseln till samtliga kopparkapslar var avstängd två veckor under december 2004.

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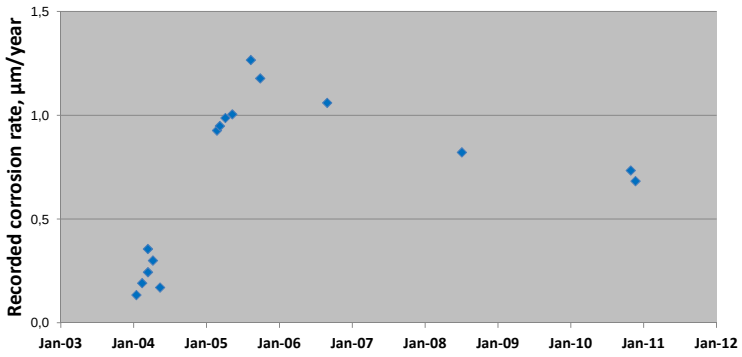
Äspö HRL – Prototype Repository – dh 5
Recorded corrosion rate on copper electrodes during 2004-2010



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Before retrieval of copper electrodes

- Perform another measurement of the corrosion potentials before retrieval of a bentonite block containing the copper electrodes
- Ditto SmartCET® corrosion monitoring

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Retrieval

- Locate the copper electrodes properly by means of documentation and cables
- Remove the copper electrodes in one bentonite test package, tentatively 1 dm bentonite around the electrodes
- Place the removed bentonite package in a proper package, method used by Clay Technology, and evacuate air from the package

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"Preservation" of the bentonite block

A few questions at issue:

- How much have the copper electrodes already been influenced by the opening of the outer section and the subsequent retrieval work, or are the electrodes still more or less unaffected? Changes have definitely occurred since at least part of the pressure from the surrounding bentonite and backfill has been released. *Corrosion potential measurements may be informative.*
- When the bentonite block with the copper electrodes is removed, the remaining pressure from the surrounding rock, bentonite and backfill will be released. This resulted in an increased corrosion rate and change of the corrosion potential for the copper electrodes from a LOT test parcel A2 during retrieval. *Thus, both SmartCET® corrosion monitoring, to obtain corrosion rates, and corrosion potential measurements have been proposed for some time after the retrieval to find out and compare the behaviour with the findings from the LOT test package.*
- Alternatives for "preservation": (i) we trust that the surrounding bentonite give adequate protection for a while, (ii) we improve the protection by means of moulding the bentonite block in paraffin, or (iii) we select a method that will definitely protect the copper electrodes from any influence of the surrounding air. Preparation for this has to be performed in advance, in spite of the fact that the electrodes may have experienced a substantial influence already.

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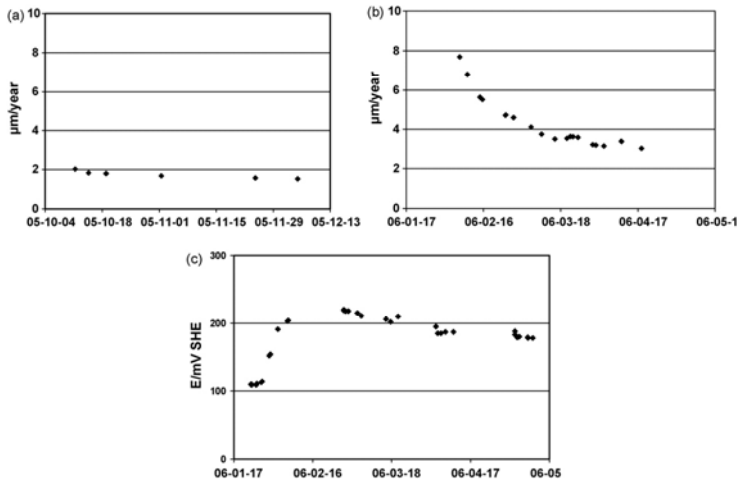
The recorded corrosion rate: (a) just before retrieval and (b) after retrieval, and (c) the corrosion potential of one of the pre-exposed copper electrodes after retrieval.



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Anticipated environmental conditions in the bentonite block after retrieval

- After retrieval the conditions for the copper electrodes in the removed bentonite package are anticipated to be quite similar but of course not the very same as before, since the pressure on the bentonite block in the rock has been released.
- Temperature
- External environment

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Electrochemical tests immediately after retrieval

- Install reference electrodes in the bentonite block
- Measure and follow corrosion potentials
- Perform SmartCET® corrosion monitoring
- Perform electrochemical impedance spectroscopy
- Compare with earlier experiences from retrieval of the bentonite test package from LOT test parcel A2

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Start of post-test examination

- Remove one of the outer copper electrodes
- Decision: Select either **Black** or **Red**!
- Step-by-step removal
- Photo documentation during removal
- Control localisation and cable colour
- Careful photo documentation of the electrode surface
- Cut the electrode in pieces for further examination

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Tentative content of post-test examination

- Determine corrosion products by XRD
- Examine cross-sections
- Examine and document possible localisation of the corrosion attack
- Examine further “question-marks” on the electrode surface
- Use laser scanning confocal microscopy for “profilometry”
- Copper profiles in the bentonite; freeze dry (method used by Clay Technology)
- others

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Within reach

- The corrosion rate from 2004 through 2010
  - maximum possible corrosion rate (conservative approach)
  - supporting work in progress for better estimate (measuring frequency, other redox reactions, ...)
- Corrosion end products
  - XRD
  - Raman spectroscopy
- Corrosion appearance
  - photography
  - microscopy
- Corrosion product

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Unattainable

- Corrosion potential from emplacement up to 2011-02-04
  - not measured, have to rely on modelling and supporting work
- Average corrosion rate from gravimetry

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SmartCET® corrosion monitoring systems

have been used with three-electrode set-ups and nominally identical Electrodes. The systems operate in a multi-technique mode continuously cycling through the measurements in order as follows:

- electrochemical noise (300 s),
  - with data taken at a frequency of 1 Hz
- **polarization resistance and harmonic distortion analysis** (100 s),
  - 50 mV peak-to-peak voltage perturbation amplitude
  - 10^{-2} Hz voltage perturbation frequency
  - current response measured and analysed synchronously
- and solution resistance (30 s).

Thus, records are obtained about every 7 min.

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Comparison...

- **Från annan presentation**

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Information from ... LOT A2

- **Från annan presentation**

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