

Appendix 3

Post-test examination of copper coupons from LOT test parcel A2 regarding corrosion

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Post-test examination of copper coupons from LOT test parcel A2 regarding corrosion

Report compiled by Bo Rosborg based upon experimental work performed at Clay Technology AB, Stockholm University and Studsvik Nuclear AB.

Summary

Coupons of pure copper have been exposed in LOT test parcel A2 at the Äspö Hard Rock Laboratory from October 1999 to January 2006. The conditions have been similar to those in a KBS-3 repository. This report documents the post-test examination of the copper coupons with the objective to determine the nature and extent of the corrosion.

A brownish corrosion product layer with blue-green corrosion products here and there on top of it covered most of the surface. Cuprite and paratacamite were identified as the major corrosion products.

As before the nature of the corrosion can be classified as a somewhat uneven general attack. A number of surface defects were found in the copper coupons, which are believed to originate from the manufacturing rather than being a result of corrosion. Any obvious signs of pitting cannot be claimed.

The average corrosion rate was estimated to be less than 0.5 $\mu\text{m}/\text{year}$.

Copper was found to penetrate 500 μm into the bentonite.

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Background information

The test series “Long Term Test of Buffer Material” (LOT) has been initiated at the Äspö Hard Rock Laboratory with conditions similar to those in a KBS-3 repository (1). The main purpose is to study the behaviour of the bentonite clay. Wyoming bentonite with the commercial name MX-80 has been used. However, additional testing has been included, and the post-test examination of copper coupons exposed in bentonite is the subject of this report.

Objective: Determine nature and extent of copper corrosion.

Attempt: “Quantitative information about the mean corrosion rate. Qualitative information about pit corrosion and corrosion products.” (2)

Hypothesis: The average corrosion rate is less than 7 μm per year.

In LOT test parcel A2 two coupons each of pure copper were as before exposed in bentonite rings 22 and 30 respectively (3). Furthermore three cylindrical copper electrodes for real-time corrosion monitoring were also exposed in bentonite ring 36 (4). The latter are not treated in this report.

Background information about the copper coupons is found in Appendix A. The nominal dimensions of the coupons were 60 x 15 x 1.5 mm. They were manufactured by milling, and then one side was slightly polished. Test parcel A2 was emplaced on October 29, 1999. Full temperature lasted from September 2000 up to December 5, 2005. The test parcel was retrieved on January 16, 2006. The bentonite rings A222 and A230 have been exposed at temperatures of about 75°C and 30°C respectively. The total time of exposure is more than 6 years, and the time of exposure at full temperature is 5 years and 3 months.

Additional groundwater from a dedicated bore-hole in the nearby tunnel wall was added through a small titanium filter tip in the upper part of the test parcel. Full saturation was expected after less than one year at free access to water in the rock/bentonite interface. The actual full water saturation was reached within two years as interpreted from measured moisture and pressure results.

After cutting apart the bentonite rings from the test parcel at Äspö on 2006-01-17/19, they were immediately wrapped in plastic sacks which were air evacuated by means of a vacuum pump. They were then transported to Studsvik and stored in their plastic sacks until further work. The copper coupons in bentonite ring A222 were unfortunately damaged by the cutting wheel during retrieval, thus making an accurate estimation of the corrosion rate for these coupons impossible.

Copper coupons from two test series have earlier been examined. The average corrosion rate of a copper coupon in LOT pilot test parcel S1 (exposed at about 50°C) was estimated to less than 3 μm per year (5). In LOT test parcel A0 the average corrosion rate of copper coupons (exposed at 35°C and about 80°C respectively) was estimated to less than 4 μm per year (6). Any obvious signs of pitting were not found.

Three test parcels are still exposed and will be retrieved later (2).

This report forms an input to the final report concerning the evaluation of the exposure of LOT test parcel A2 to be compiled by Ola Karnland, Clay Technology AB.

Experimental procedure

Copper coupons A222E and A222F were clearly seen on the side of bentonite ring A222 due to the unfortunate damage by the cutting wheel. Thus, it was quite trivial to cut apart the bentonite ring close to the coupons and break loose the coupons. Coupons A230G and A230H were removed from bentonite ring A230 by a step-wise “fracturing” of the bentonite ring until one end of each coupon was spotted. Then the bentonite piece containing the coupon was prepared to facilitate the breaking loose of the coupon.

For the sequence of actions performed during investigation of copper coupons A230G and A230H, see Appendix B. Coupons A222E and A222F were less extensively investigated. It was intended to incorporate a reference coupon in the present investigation through all cleaning procedures for the purpose of comparison. However, due to the fact that two out of four copper coupons in the test parcel were damaged during retrieval, it was decided to save the reference coupons for later investigations of copper coupons within the LOT project.

Sampling of corrosion products was performed immediately after breaking loose each copper coupon. The samples and the copper coupons were stored in a desiccator until further work.

All coupons but for coupon A222F were ultrasonically descaled in 10 % H_2SO_4 solution before final microscopy (7).

Corrosion products

After breaking loose a copper coupon from the bentonite, corrosion products could as found before (5-6) be seen on parts of the bentonite surfaces facing the copper coupon, see Figure 1.1, thus revealing a better adherence to the bentonite on part of the surface. A brownish corrosion product layer with blue-green corrosion products here and there on top of it covered most of the copper coupons, see Figure 1.2. However, apparently bare copper surface was also seen. The blue-green corrosion products revealed the presence of bivalent copper.



Figure 1.1 Corrosion products seen on the adjacent bentonite after breaking loose copper coupon A230H. (The copper coupon is in place in the upper bentonite piece.)

Scraping off the brownish corrosion product layer on the bentonite revealed blue-green corrosion products below. X-ray diffraction (XRD) has confirmed that the main constituent of the brownish layer is cuprite and that the blue-green corrosion products contain paratacamite.

Appendix C shows the extent of the XRD and the energy dispersive spectroscopy (EDS) work.

The blue-green corrosion products were not only unevenly distributed on the copper coupons, but also in between them. Copper coupon A230G contained the most, see Figure 1.2 through 1.5. (It showed also the maximum weight loss, see Appendix E.) The EDS data revealed that the blue-green corrosion products penetrated into the bentonite.

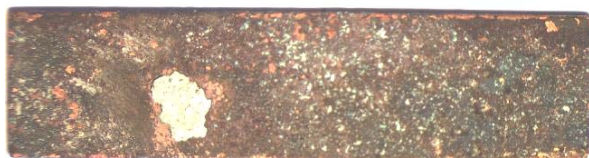


Figure 1.2 Distribution of the blue-green corrosion products (almost white in the picture) on side 1 of copper coupon A230G. (The nominal dimensions of the coupon are 60 x 15 x 1.5 mm.)

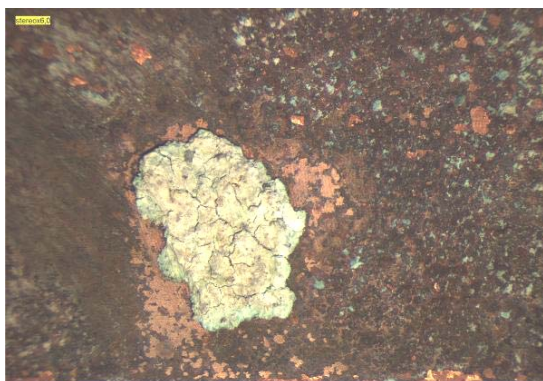


Figure 1.3 Part of Figure 1.2 with the blue-green “crust”.

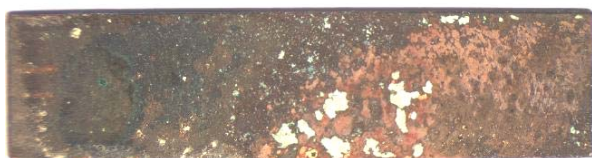


Figure 1.4 Distribution of the blue-green corrosion products (almost white in the picture) on side 2 of copper coupon A230G.

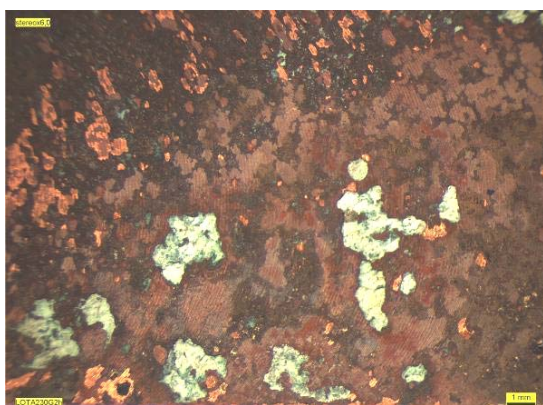


Figure 1.5 Part of Figure 1.4 with the blue-green corrosion products.

Microscopy

The extent of the microscopy is apparent from Appendix D.

As before (6) the micro hardness indentation marks on the polished side of coupons A222E and A230G made before exposure, as shown in Figure 2.1, were not found after exposure, in spite of the fact that the identification, the milling cutter and the polishing marks are clearly seen (and that the average corrosion rate is estimated to be less than $0.5 \mu\text{m}/\text{year}$, see Appendix E).

Since the indentation marks were not found a series of photos were as one alternative taken in the centre of the coupon, see Figure 2.2. As another alternative in order to visualize the extent of the corrosion attack, or rather the absence of any major corrosion, photos were also taken of the identification marks (the letters), see Figure 2.3.

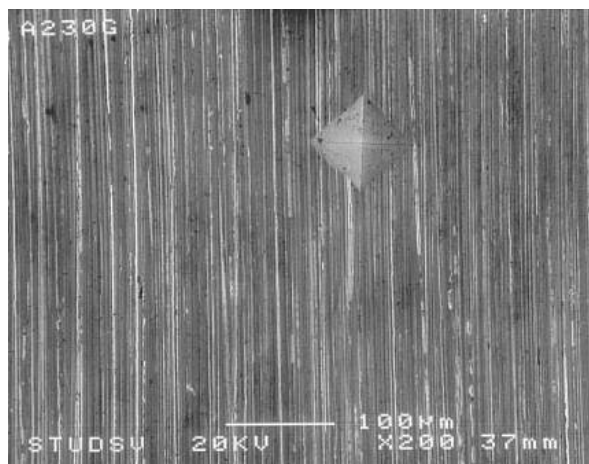


Figure 2.1 The micro hardness indentation mark (a Vickers pyramid indent) in the centre of the slightly polished side 1 of copper coupon A230G before exposure.

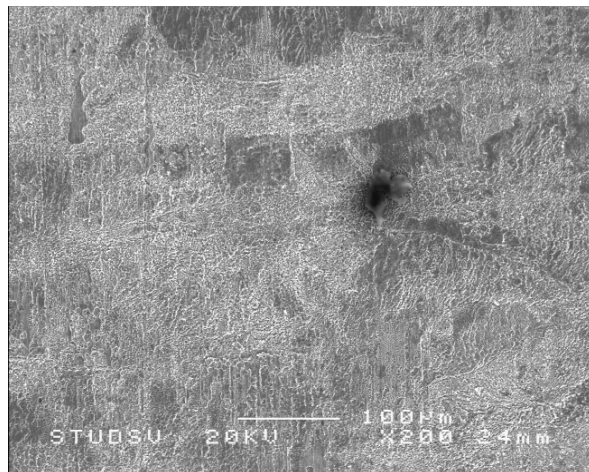


Figure 2.2 The centre of the slightly polished side 1 of copper coupon A230G after exposure. (The intention was to find the indentation mark shown in Figure 2.1 after exposure and take a picture of the very same area. However, the mark was not found.)

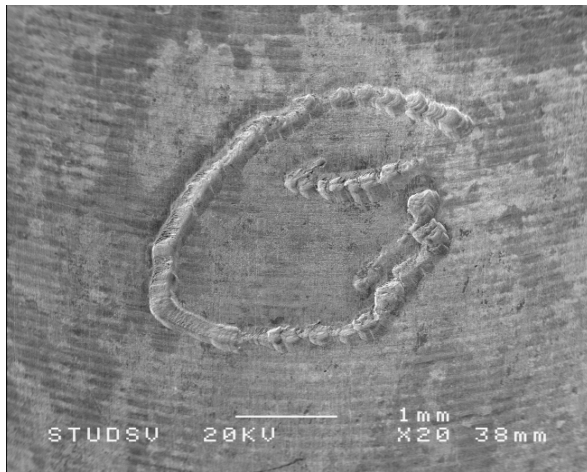


Figure 2.3 The identification mark (the letter G) on side 1 of copper coupon A230G after exposure. (Do observe that the milling cutter marks, but not the polishing marks, are clearly seen.)

A number of surface defects of the kind shown in Figure 2.4 were, however, found in the copper coupons. They are believed to originate from the manufacturing rather than being a result of corrosion. Any signs of active pits could not be found.

Figures 2.7 through 2.9 show the area below and next to the crust in Figure 1.2 after descaling. The amount of corrosion is somewhat higher at the crust. However, any unambiguous signs of pitting are not obvious.

In summary, the nature of the corrosion can as before be classified as a somewhat uneven general attack. Any obvious signs of pitting cannot be claimed.

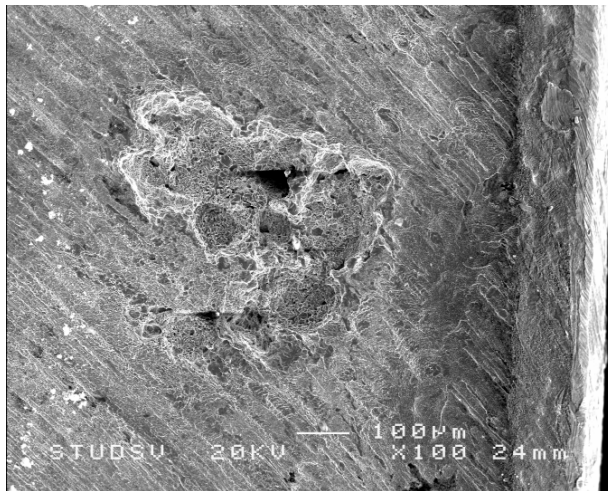


Figure 2.4 Surface defect found in copper coupon A230G close to one edge.

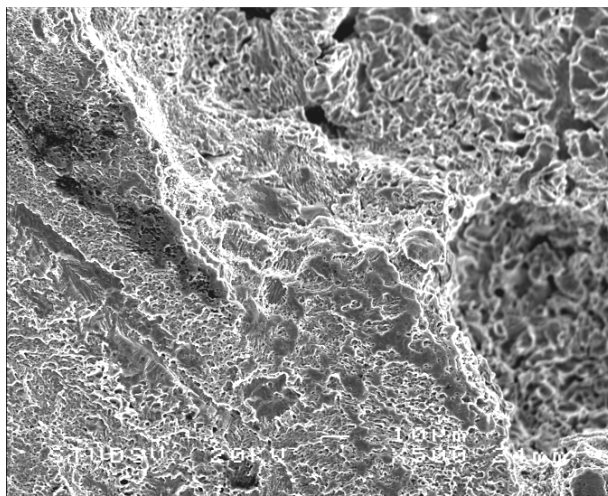


Figure 2.5 Detail of Figure 2.4.

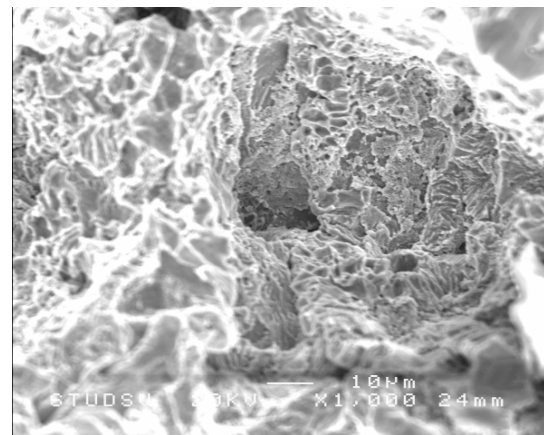
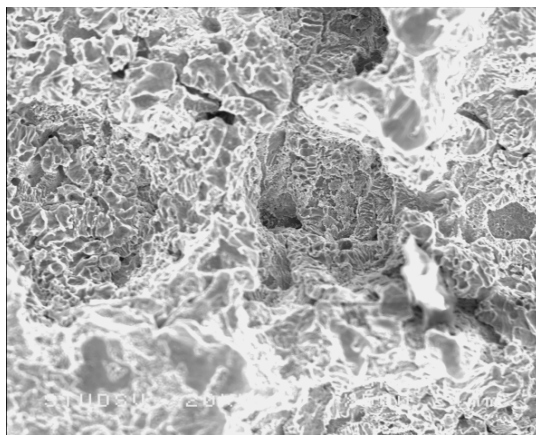


Figure 2.6 Another detail of Figure 2.4 in two magnifications.

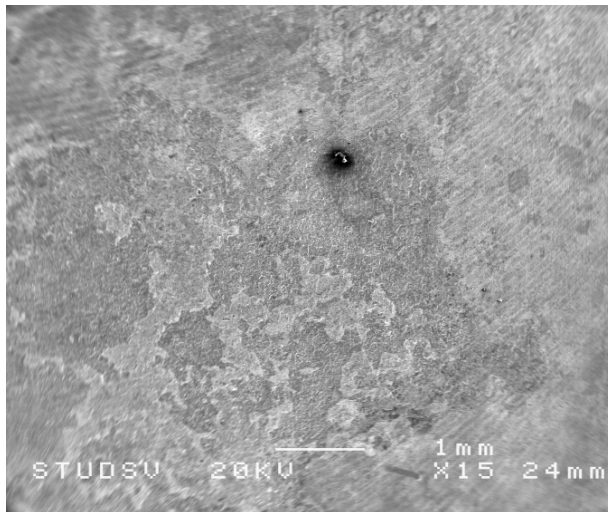


Figure 2.7 The area at the crust in Figure 1.2 after descaling.

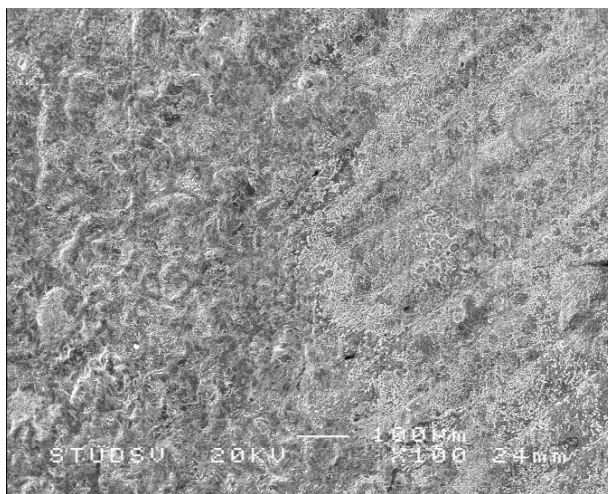


Figure 2.8 Detail of Figure 2.7 showing the boundary between more corroded and less corroded area (the latter to the right where the milling cutter marks are clearly seen).

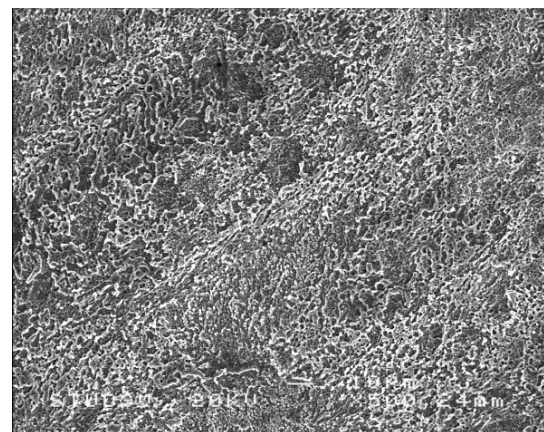
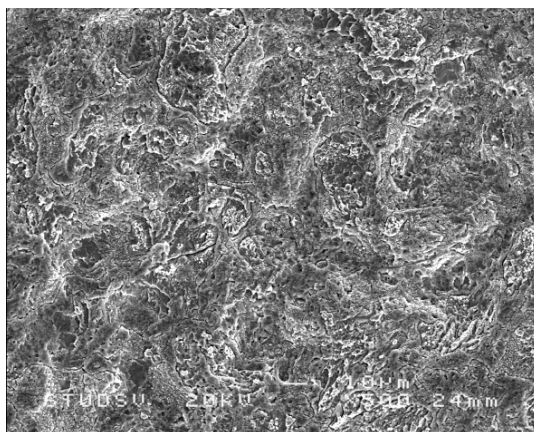


Figure 2.9 The more corroded area (to the left) and the less corroded area (to the right) in higher magnification.

Gravimetry

The weight loss data are compiled in Appendix E. Based upon the maximum weight loss obtained (that is for copper coupon A230G) the average corrosion rate of copper was estimated to less than 0.5 $\mu\text{m}/\text{year}$.

The estimated average corrosion rate is considerably lower than the values earlier obtained from LOT test parcels A0 and S1. The weight losses for the copper coupons in test parcel A2 (27-46 mg), exposed more than six years, are in fact lower than the weight losses for the coupons in test parcels A0 and S1 (77- 86 mg), exposed less than two years (5-6).

The estimated average corrosion rate does not contradict the earlier findings from the performed real-time corrosion monitoring in LOT test parcel A2 (4).

Penetration depth

The penetration depth of copper into the bentonite next to copper coupon A230H has been examined. The bentonite samples were prepared by means of breaking the bentonite pieces facing the coupon in such a way that fracture surfaces perpendicular to the coupon were obtained. Bentonite sample H4b was picked for EDS and the results are found in Appendix F.

Copper was found to penetrate 500 μm into the bentonite.

Acknowledgements

The contributions from Martin Birgersson, Ola Karnland, Ulf Nilsson and Siv Olsson, at Clay Technology AB, Lars Göthe (XRD) at Stockholm University, and Eva Sund and Roger Lundström (SEM and stereomicroscopy) at Studsvik Nuclear AB are gratefully acknowledged.

References

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2. O Karnland and T Sandén, Long term test of buffer material, Installation report phase II, Clay Technology AB, Lund, September 2001.
3. B Rosborg, Efterundersökning av kopparkuponger från LOT testpaket A2 avseende korrosion, Svensk Kärnbränslehantering AB, 2006-01-11 (AP TD F62-06-012).
4. B Rosborg et al, Real-time monitoring of copper corrosion at the Äspö HRL, Proc 2nd Inter Workshop Prediction of Long Term Corrosion Behaviour in Nuclear Waste Systems, Nice, September 2004, p 11-23.
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6. B Rosborg, LOT – Investigation of copper coupons from test parcel A0, 2002-10-02.
7. B Rendahl, Avlägsnande av korrosionsprodukter på koppar (utkast II 98-08-18), Korrosionsinstitutet, Stockholm, 1998 (KI Rapport 65 221).

Appendix A. Copper coupons

[illegible]

Appendix B. Sequence of actions

Sequence of actions during evaluation of the copper coupons

Start

- Bentonite rings 22 and 30 containing the copper coupons were received at Äspö
- The bentonite rings were stored in the received packages at Studsvik until start of examination of the coupons
- Reference copper coupons are stored in a desiccator at Studsvik

Procedure

- Photographing of bentonite rings 22 and 30 as received
- Break loose a copper coupon
- Record the position of the coupon in the bentonite block
- Photographing of the coupon and adjacent bentonite pieces
- Photographing of the coupon in a stereomicroscope
- Weighing of the coupon after spraying with pure alcohol
- Perform either SEM/EDS or scrape off corrosion products from the coupon
- Store the coupon in a desiccator until further cleaning and continued examination
- (For examination of corrosion products, see main text)
- SEM (loose corrosion products and corrosion attack)
- Ultrasonic cleaning in pure alcohol during 1 min
- Drying and weighing
- Photographing
- Ultrasonic cleaning in pure alcohol during 4 min
- Drying and weighing
- Photographing
- Ultrasonic cleaning in pure alcohol during another 5 min
- Drying and weighing
- Photographing
- SEM (adherent corrosion products and corrosion attack)
- Ultrasonic treatment in 10 % H₂SO₄ solution for 5 min
- Drying and weighing
- Another ultrasonic treatment in 10 % H₂SO₄ solution for 5 min
- Drying and weighing
- Microscopy (for final verification of corrosion attack)

Corrosion products on the coupon

See Appendix C.

Corrosion products in the bentonite

See also Appendix F.

- Break a bentonite piece facing a copper coupon to obtain a cross-section perpendicular to the coupon
- Freeze-drying of the bentonite samples at Clay Technology
- SEM/EDS on the cross-section (penetration of copper into the bentonite)

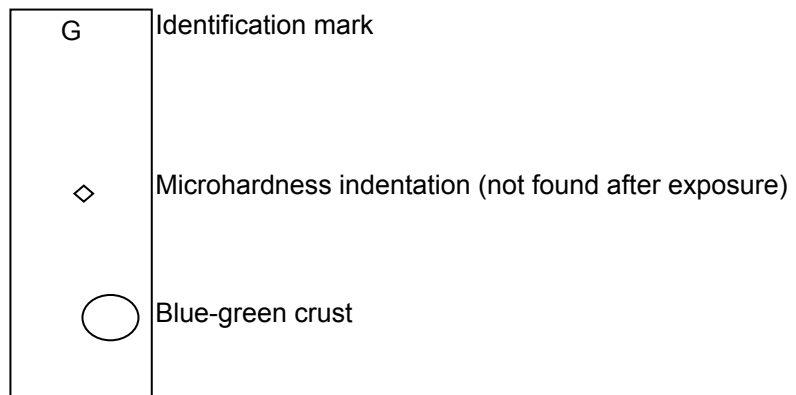
Appendix C ID 1. Corrosion products

Work performed at Studsvik on 2006-05-18.

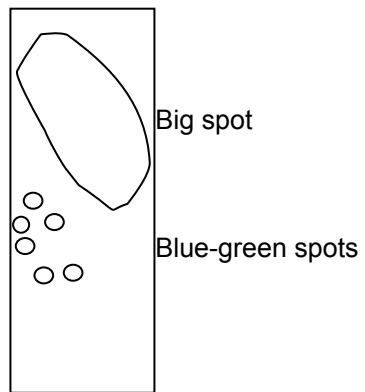
Cu-K, acceleration voltage 20 kV, live time 100 s

[illegible]

Copper coupon A230G side 1



Copper coupon A230G side 2



Visual observations

Work performed at Clay Technology and at Studsvik.

Corrosion products were found not only on the copper coupon, but also on the bentonite, after breaking loose the specimen. Thus, revealing a better adherence to the bentonite on part of the surface.

Blue-green corrosion products were present on part of the surface. Thus revealing the presence of bivalent copper.

The blue-green corrosion products were unevenly distributed on the copper coupon; spots and a "crust" (on A230G side 1).

Findings from XRD

Identified corrosion products:	cuprite	Cu ₂ O
	paratacamite	Cu ₂ (OH) ₃ Cl
Not found:	malachite	Cu ₂ CO ₃ (OH) ₂
	nantokite	CuCl
	tenorite	CuO

Findings from EDS

Higher Al, Si, Cl and Fe contents found in the blue-green corrosion products on copper coupon A230G compared to the adjacent surface on the coupon. (Also lower Cu contents compared to the nextto analyses.)

Major Al and Si contents in the blue-green corrosion products, reveal that the blue-green corrosion products penetrate the bentonite.

The increased Cl content can be explained by the presence of paratacamite in the corrosion products.

That Fe is detected in the blue-green corrosion products, but not next to them (in 3 out of 3 cases), can also be explained by the presence of bentonite in the corrosion products. (See the spectra in ID 4. Penetration depth.)

Appendix E ID 3. Gravimetry

ID 3. Gravimetry

Weight changes of copper coupon A222E

Work performed at Studsvik on 2006-05-18

Scale: Mettler AE163, calibrated 2005-10-18

Note: The coupon was unfortunately damaged during retrieval in Äspö; the cutting wheel removed part of the coupon when separating bentonite rings A223 and A222. Thus, only an estimate of the weight loss can be obtained.

Condition	Weight	Photo
Original weight	12,0564 g	
<i>Coupon unfortunately damaged during retrieval in Äspö</i>		1699
After removal from bentonite ring A222 and spraying with alcohol	11,0349	
After 5 min ultrasonic treatment in 10 % H ₂ SO ₄ solution	11,0090	
After 10 min ditto	11,0070	
After 15 min ditto	11,0054	
<i>Weight loss 11,0349-11,0070=</i>	<i>27,9 mg</i>	
<i>Estimate of total weight loss 12,0564/11,0070*27.9=</i>	<i>30,6 mg</i>	

Weight changes of copper coupon A222F

Work performed at Studsvik on 2006-05-18

Scale: Mettler AE163, calibrated 2005-10-18

Note: The coupon was unfortunately damaged during retrieval in Äspö; the cutting wheel removed part of the coupon. Thus, only an estimate of the weight loss can be obtained.

Condition	Weight	Photo
Original weight	12,0856 g	
<i>Coupon unfortunately damaged during retrieval in Äspö</i>		1699
After removal from bentonite ring and spraying with alcohol	9,0147	

Coupon left for possible later cathodic reduction of part of the remaining surface and indirect estimation of the corrosion rate.

Weight changes of copper coupon A230G

Work performed at Studsvik on 2006-05-16 and 2006-05-18

Scale: Mettler AE163, calibrated 2005-10-18

Condition	Weight	Photo	Note
Original weight	12,1411 g		
After removal from bentonite ring	12,1642	1796-1801	
After spraying with alcohol	12,1487		
After 1 min ultrasonic cleaning in alcohol	12,1449		
After 5 min ditto	12,1356		
After 10 min ditto	12,1317		Sample loose corrosion products, see ID 1
After 5 min ultrasonic treatment in 10 % H ₂ SO ₄ solution	12,0953		
After 10 min ditto	12,0952		
Total weight loss	45,9 mg		

Weight changes of copper coupon A230H

Work performed at Studsvik on 2006-04-12

Scale: Mettler AE163, calibrated 2005-10-18

Condition	Weight	Photo
Original weight	11,7696 g	
After removal from bentonite ring	11,76307	1756, 1757, 1763
After spraying with alcohol	11,76154	
After 1 min ultrasonic cleaning in alcohol	11,75974	SD21+SD22
After 5 min ditto	11,75820	
After 10 min ditto	11,75607	
After 5 min ultrasonic treatment in 10 % H ₂ SO ₄ solution	11,74298	1782, 1783, 1784
After 10 min ditto	11,74264	
Total weight loss	27,0 mg	

Estimate of average corrosion rate

The average corrosion rate has been calculated from the following set of data:

Maximum weight loss	45,9	mg
Surface area	20,3	cm²
Density of pure copper	8,96	g/cm³
Total time of exposure	6,2	year
Average corrosion rate	0,41	µm/year

Average corrosion rate <0,5 µm/year

Comments

The estimated average corrosion rate is considerably lower than the values obtained from LOT test parcel A0 and S1. The weight losses for the copper coupons in test parcel A2 (27-46 mg), exposed more than six years, are in fact lower than the weight losses for the coupons in test parcels A0 and S1 (77- 86 mg), exposed less than two years.

The estimated average corrosion rate is not in conflict with the earlier findings from the performed real-time monitoring in LOT test parcel A2.

Appendix F ID 4. Penetration depth

ID 4. Penetration depth

Cu profile in bentonite next to copper coupon A230H - Bentonite sample H4b
Sampling on 2006-03-14 and freeze drying up to 2006-05-11performed at Clay Technology and EDS performed at Studsvik on 2006-05-12
JSM-6300 scanning electron microscope; Noran/Voyager EDS system
Cu-K, acceleration voltage 20 kV, live time 100 s, area 30,5 x 23,4 µm (61 x 47 mm; 20 mm =10 µm)

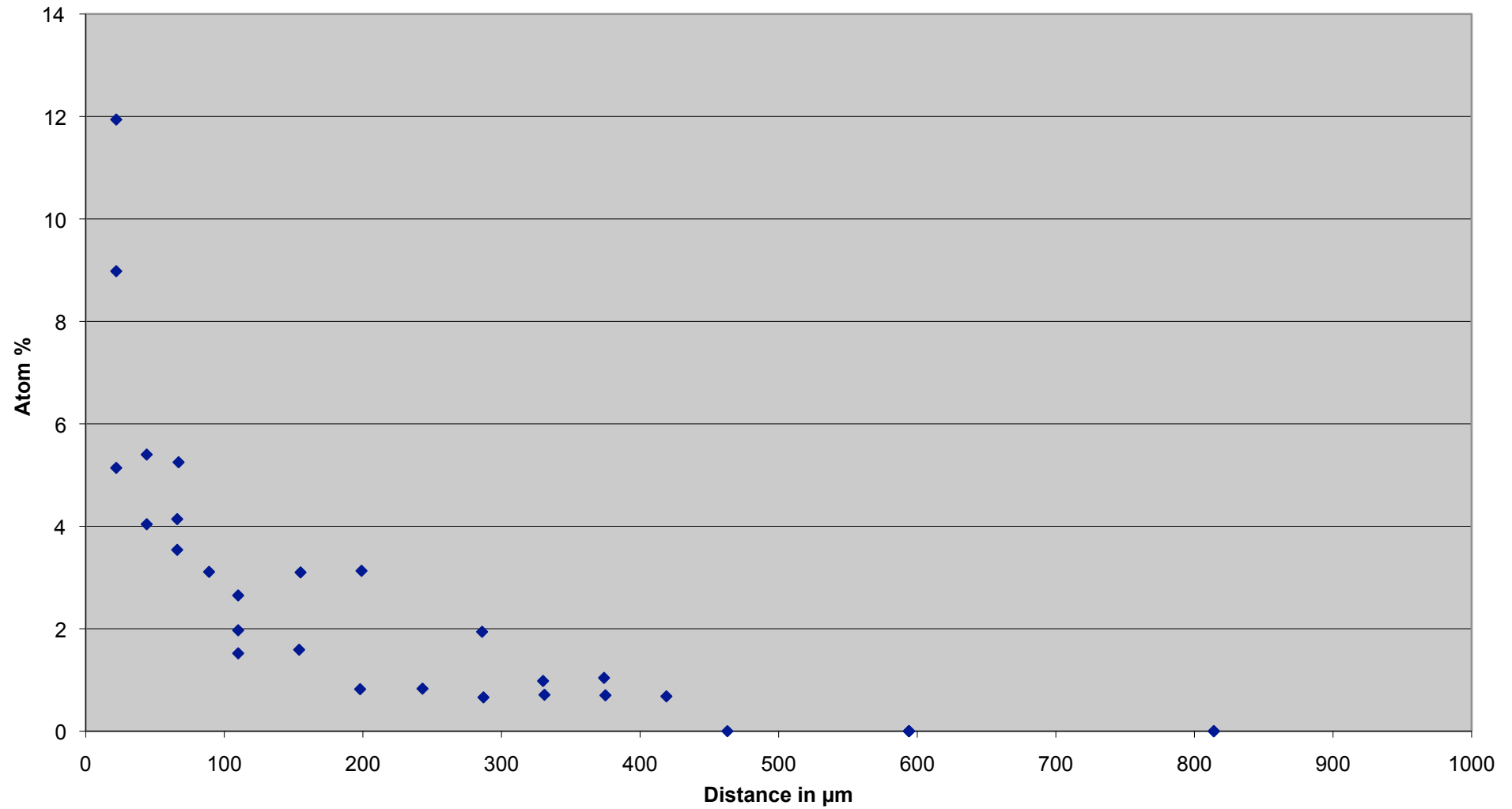
Trace	Area	Identification	Pos V	Pos H		Distance µm	Chi-sqd	Cu-K		k-ratio	ZAF	Atom %		Wt %		Cu-L		Note	Atom %										Fe-K		Atom %														
				before	after			net counts	error			value	error	net counts	error	Na	Mg		Al	Si	S	Cl	K	Ca	Fe	Cu	Au	net counts	error																
1		H4b1 surface	24,980	24,457	24,446	0																																							
	a	H4b1a	24,980	24,479		22	2,47	7054	211	0,1182	1,030	8,98	12,18	0,36		5654	169			2,05	14,99	59,88		7,20		0,84	2,08	8,98	3,98	2194	93	2,08													
	b	H4b1b	24,980	24,523		66	7,68	3040	106	0,0567	1,019	4,32	5,78	0,20		2077	154			2,07	16,55	67,88			0,52	1,68	1,91	4,32	5,08	1777	159	1,91													
	c	H4b1c	24,980	24,567		110	3,26	1085	82	0,0240	1,086	1,52	2,60	0,20		1061	103		4,42	3,28	21,79	59,16			0,26	0,99	2,59	1,52	4,11	2469	157	2,59													
	d	H4b1d	24,956	24,611		154	4,83	871	78	0,0247	1,085	1,59	2,68	0,24					3,12		21,27	61,84				4,03	3,30	1,59	4,78	2402	97	3,30													
	e	H4b1e	24,980	24,655		198	1,60	533	72	0,0127	1,069	0,82	1,36	0,18					2,63	2,44	20,54	62,55		1,26	0,41	0,95	2,77	0,82	5,64	2369	90	2,77													
	f	H4b1f	24,980	24,699		242	5,30	1450	82	0,0404	1,077	2,63	4,35	0,25		514	73			2,17	21,78	62,94				2,67	2,67	2,63	5,13	1952	149	2,67													
	g	H4b1g	24,980	24,743		286	3,26	1073	79	0,0295	1,083	1,94	3,20	0,24		425	69			2,88	21,10	60,57				0,46	1,99	4,33	1,94	4,71	3203	161	4,33												
	h	H4b1h	24,980	24,787		330	1,87	408	68	0,0146	1,056	0,98	1,54	0,26					2,76	3,04	21,50	59,37				0,53	1,16	4,07	0,98	6,58	2234	150	4,07												
	i	H4b1i	25,048	24,831		374	1,55	461	64	0,0135	1,022	1,04	1,38	0,19						1,81	15,87	67,06				0,64	1,34	4,31	1,04	5,41	2511	153	4,31												
	j	H4b1j	24,980	25,051		594	1,75	0				0	0					No counts		1,97	15,47	72,56		0,53		2,48	2,11		1,91	2461	89	2,11													
k	H4b1k	24,980	25,271		814	2,21	0				0	0					ditto		3,49	22,22	64,36				0,25	1,10	2,16		2,84	2114	147	2,16													
2		H4b2 surface	23,198	24,637	24,625	0																																							
	a	H4b2a	24,660			23	6,03	2226	150	0,0696	1,076	4,63	7,48	0,50		298	80				19,38	63,94			0,38	3,92	2,69	4,63	4,96	1732	146	2,69													
	b	H4b2b	24,682			45	5,86	3565	174	0,0991	1,080	6,51	10,70	0,52		3115	86				16,93	65,71				2,94	1,94	6,51	4,50	1428	80	1,94													
	c	H4b2c	24,704			67	1,78	2912	102	0,0788	1,103	5,25	8,69	0,30		3014	83		1,41	10,87	49,19	15,67	2,21	0,37		10,14	1,20	5,25	3,69	894	135	1,20													
	d	H4b2d	24,726			89	3,39	1737	150	0,0463	1,109	3,11	5,13	0,44		1627	77		1,82	10,45	40,50	24,51				14,55	1,22	3,11	3,84	902	75	1,22													
	e	H4b2e	24,748			111	5,12	1563	148	0,0435	1,099	2,72	4,78	0,45		2017	146		1,96	18,88	67,31					3,39	1,93	2,72	3,81	1486	141	1,93													
	f	H4b2f	24,792			155	4,53	1516	138	0,0491	1,095	3,10	5,38	0,49		983	73		1,84	15,81	69,93				0,39	3,25	1,66	3,10	4,02	1089	131	1,66													
	g	H4b2g	24,836			199	1,16	1162	77	0,0467	1,077	3,13	5,03	0,33		383	69		3,60		13,34	58,89	6,89	1,80		0,35	2,96	3,74	3,13	4,99	1854	80	3,74												
	h	H4b2h	24,880			243	2,24	555	69	0,0137	1,123	0,83	1,54	0,19						2,72	19,14	68,62				0,32	0,97	1,72	0,83	2,51	1571	138	1,72												
	i	H4b2i	24,924			287	2,11	454	67	0,0109	1,119	0,66	1,22	0,18						2,92	19,04	68,92				0,50	0,79	1,74	0,66	2,77	1632	81	1,74												
	j	H4b2j	24,968			331	2,24	430	64	0,0117	1,110	0,71	1,30	0,19		50	80			2,64	19,77	69,17					0,82	1,87	0,71	3,21	1527	138	1,87												
	k	H4b2k	25,012			375	1,61	370	61	0,0115	1,111	0,70	1,28	0,21						2,75	16,99	71,16					0,82	2,03	0,70	3,06	1451	75	2,03												
	l	H4b2l	25,056			419	3,39	385	60	0,0113	1,121	0,68	1,26	0,20						2,84	18,18	68,35					2,81	1,81	0,68	2,62	1388	75	1,81												
	m	H4b2m	25,100			463	2,06	0				0	0				No counts		2,90	20,38	66,86					1,77	1,86		3,07	1519	77	1,86													
	n	H4b2n	25,231			594	2,55	0				0	0				ditto		3,38	18,45	69,17				0,41	1,76	1,49		2,60	1314	133	1,49													
	o	H4b2o	25,451			814	1,75	0				0	0				ditto		2,73	16,25	72,31		0,48	0,32		1,23	1,46		2,79	1160	133	1,46													
p	H4b2p	26,265			1628	1,49	0					0	0				ditto		2,69	18,08	56,81	9,48				6,75	2,08		1,41	1326	72	2,08													
3		H4b3 surface	26,762	24,283	24,275	0																																							
	a	H4b3a	24,305			22	4,25	3160	105	0,1408	0,974	11,94	13,71	0,46		832	84		2,49	16,72	51,32						1,30	3,01	11,94	13,21	1038	136	3,01												
	b	H4b3b	24,327			44	1,78	1726	153	0,0554	1,023	4,04	5,66	0,50		611	67		1,94	19,18	60,10		2,30			1,03	2,47	4,04	9,94	1372	78	2,47													
	c	H4b3c	24,349			66	2,13	1739	167	0,0403	0,968	3,54	3,91	0,38		393	68		2,12	15,01	60,78		2,28	0,33		1,71	2,75	3,54	11,46	1776	88	2,75													
	d	H4b3d	24,393			110	1,29	461	66	0,0199	0,934	1,97	1,86	0,27					2,20	15,42	55,82				0,57	1,15	3,84	1,97	16,97	1139	69	3,84													
4		H4b4 surface	27,982	24,178	27,982	0																																							
	a	H4b4a	24,200			22	3,85	2223	164	0,0633	1,001	5,14	6,33	0,47		733	62		2,02	15,37	62,42				1,00	3,64	3,07	5,14	7,35	1740	84	3,07													
	b	H4b4b	24,222			44	4,05	2408	97	0,0780	1,053	5,40	8,21	0,33		1734	120		2,18	20,63	58,00			0,63	4,15	2,60	5,40	6,40	1536	148	2,60														
	c	H4b4c	24,244			66	4,17	2116	155	0,0616	1,064	4,14	6,56	0,48		1219	76		1,80	21,44	60,50					3,97	2,40	4,14	5,75	1623	149	2,40													
	d	H4b4d	24,288			110	2,91	1140	81	0,0399	1,067	2,65	4,25	0,30		392	66		1,80	21,46	63,10					2,26	2,99	2,65	5,74	1708	84	2,99													

Cu profile in bentonite next to copper coupon A230H - Bentonite sample H4b

Superimposed data. Only data with chi-sqd <5 included.

Trace	Identification	Pos V	Pos H before	Distance µm	Chi-sqd	Cu-K		k-ratio	ZAF	Atom %	Wt %		Cu-L		Note
						net counts	error				value	error	net counts	error	
1	H4b1a	24,980	24,479	22	2,47	7054	211	0,1182	1,030	8,98	12,18	0,36	5654	169	
3	H4b3a	26,762	24,305	22	4,25	3160	105	0,1408	0,974	11,94	13,71	0,46	832	84	
4	H4b4a	27,982	24,200	22	3,85	2223	164	0,0633	1,001	5,14	6,33	0,47	733	62	
3	H4b3b	26,762	24,327	44	1,78	1726	153	0,0554	1,023	4,04	5,66	0,50	611	67	
4	H4b4b	27,982	24,222	44	4,05	2408	97	0,0780	1,053	5,40	8,21	0,33	1734	120	
3	H4b3c	26,762	24,349	66	2,13	1739	167	0,0403	0,968	3,54	3,91	0,38	393	68	
4	H4b4c	27,982	24,244	66	4,17	2116	155	0,0616	1,064	4,14	6,56	0,48	1219	76	
2	H4b2c	23,198	24,704	67	1,78	2912	102	0,0788	1,103	5,25	8,69	0,30	3014	83	
2	H4b2d	23,198	24,726	89	3,39	1737	150	0,0463	1,109	3,11	5,13	0,44	1627	77	
1	H4b1c	24,980	24,567	110	3,26	1085	82	0,0240	1,086	1,52	2,60	0,20	1061	103	
3	H4b3d	26,762	24,393	110	1,29	461	66	0,0199	0,934	1,97	1,86	0,27			
4	H4b4d	27,982	24,288	110	2,91	1140	81	0,0399	1,067	2,65	4,25	0,30	392	66	
1	H4b1d	24,956	24,611	154	4,83	871	78	0,0247	1,085	1,59	2,68	0,24			
2	H4b2f	23,198	24,792	155	4,53	1516	138	0,0491	1,095	3,10	5,38	0,49	983	73	
1	H4b1e	24,980	24,655	198	1,60	533	72	0,0127	1,069	0,82	1,36	0,18			
2	H4b2g	23,198	24,836	199	1,16	1162	77	0,0467	1,077	3,13	5,03	0,33	383	69	
2	H4b2h	23,198	24,880	243	2,24	555	69	0,0137	1,123	0,83	1,54	0,19			
1	H4b1g	24,980	24,743	286	3,26	1073	79	0,0295	1,083	1,94	3,20	0,24	425	69	
2	H4b2i	23,198	24,924	287	2,11	454	67	0,0109	1,119	0,66	1,22	0,18			
1	H4b1h	24,980	24,787	330	1,87	408	68	0,0146	1,056	0,98	1,54	0,26			
2	H4b2j	23,198	24,968	331	2,24	430	64	0,0117	1,110	0,71	1,30	0,19	50	80	
1	H4b1i	25,048	24,831	374	1,55	461	64	0,0135	1,022	1,04	1,38	0,19			
2	H4b2k	23,198	25,012	375	1,61	370	61	0,0115	1,111	0,70	1,28	0,21			
2	H4b2l	23,198	25,056	419	3,39	385	60	0,0113	1,121	0,68	1,26	0,20			
2	H4b2m	23,198	25,100	463	2,06	0				0	0				No counts
1	H4b1j	24,980	25,051	594	1,75	0				0	0				ditto
2	H4b2n	23,198	25,231	594	2,55	0				0	0				ditto
1	H4b1k	24,980	25,271	814	2,21	0				0	0				ditto
2	H4b2o	23,198	25,451	814	1,75	0				0	0				ditto
2	H4b2p	23,198	26,265	1628	1,49	0				0	0				ditto

Copper profile in bentonite next to copper coupon A230H



Copper profile in bentonite next to copper coupon A230H

