

## **A geochemical normalization procedure for bricks in historical archaeology**

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### **Abstract**

A reconnaissance geochemical study of 50 elements in 8 bricks of the 17<sup>th</sup> to early 20<sup>th</sup> centuries from both sides of the Atlantic (Caribbean, Hudson Valley, Denmark, Holland) was undertaken to see whether geochemistry and/or petrography might be informative about such questions as geographic source provenance, manufacture, or subsequent environmental exposure and degradation of bricks. We find that our bricks persistently preserve the geochemistry of average upper continental crust from which they are derived by weathering into clay and sand components. These materials are recombined and thermally consolidated during brick manufacture. Even badly degraded bricks preserve the “upper crust” signal. “Upper crust” normalization of brick chemical data provides a useful baseline from which to look for detailed signals displayed by particular bricks. Pre-filtering geochemical analyses of bricks through “upper crust” normalization can give statistical techniques like cluster and principal component analysis more focus by deemphasizing some of the intrinsic covariations that are not related to source material provenance, manufacture, and degradation. Individual geochemical and petrographic signals are found in our 8 bricks to be recognizably related to the heavy mineral accessories they carry, but the study is not yet sufficiently informed to reliably interpret them for provenance. The petrography and chemistry of distinctive entrained particles appears to be a more promising discriminant of source material provenance than does bulk geochemistry. Future, more detailed, studies revealing more comprehensive baseline information may change this conclusion in favor of detailed geochemistry.

### **Introduction**

As a result of an enquiry from William A. Taylor, Architect, resident of St. Croix, Virgin Islands, we undertook an examination of 8 bricks. Our object was to discover whether there were useful markers of provenance in the chemistry and petrography of the bricks presented for study. The immediate question was whether an old fort on St. Croix, Fort Sint Kruis, could be attributed to the earliest Dutch colonials, to the Spanish, or to the later Danes, all possible suspects in the fort’s construction. A preliminary examination of stone hand-specimens from blocks in the fort’s foundation suggested that they were locally sourced rather than exotic materials imported for construction, perhaps from the country of origin of the builders of the fort. Instead, anyone could have quarried those stones locally. An alternate material, brick, was known to be ‘imported’ as ballast in trans-Atlantic voyages of the 17<sup>th</sup> century and Dutch brick was known to have been used in Landhuis Santa Barbara construction on Curacao [Dr. Charles Gehring, see appendix] and there was some brickwork in Fort St. Kruis. 16<sup>th</sup> and 17<sup>th</sup> century,

new world brick-making at Jamestown and Roanoke Island has been documented by Harrington (1950, 1967), but we are unaware of such activity in the Caribbean. Gurcke (1987) claims the Jamestown, Va. bricks were in sufficient production by 1621 to export to Bermuda. At least some small ocean island communities neighboring the American coast imported bricks, rather than manufacturing them locally. Therefore attention devolved to the bricks of Ft. Sint Kruis to see whether they might reveal specific sources on the basis of their geochemistry and petrography. If the clays and other raw materials of the Rhine delta in Holland and Belgium were different from those in Denmark or North America, perhaps bricks from those different sources could be distinguished. But what determinants are useful as potential provenance markers? Our colleague Prof. Sidney Hemming has had considerable success using the Nd isotopic composition of rock fragments found in the Heinrich event pebbles of drop stone in Dansgaard-Oeschger Layers (Hemming, 2004). These layers in the sedimentary record of the North Atlantic represent the debris from the melting of icebergs released during the breakdown of the major ice sheets of the Pleistocene. The issue of pebble provenance was important for determining whether the source of the glacial debris was Laurentide or European. These alternate sources have bedrocks of sufficiently different Nd isotope composition by virtue of their disparate ages to be a useful determinant of the alternative American or European provenances. And the bedrock pebbles preserve their original source Nd isotopic composition. We decided not to go this route first for several reasons. Bricks are not pebbles. Their ingredients are clays from the end-stage of weathering that may not preserve their source rocks' Nd isotopic composition the way unweathered pebbles have been shown to do. The brick-making process of blending clays with additional possibly unrelated solid sand-sized debris for tempering and other exotic ingredients like lime, coal, barium and lead, and then firing to reconstitute as a hard material may also perturb the isotopic composition in ways not yet understood. And Nd isotopes provide a single piece of information rather than a range of potential discriminants. We therefore undertook to use LAICPMS [laser ablation inductively couple plasma mass spectrometry] to analyze the bricks for 50 major, minor, and trace elements. In addition to our quest for discriminants of geographic provenance of bricks, we were also interested in whether there might be other things of interest in the broad-brush survey of a few bricks from widely separated sources. Do the element abundances and their ratios tell us more about the source materials and their combination, or about the brick-making process and subsequent environmental degradation, or about the scale of heterogeneity sampled in the manufacture and subsequent analysis. These are open questions in the relatively limited literature on the subject of which we are aware. (Gilbert et al., 1993; López-Arce et al., 2003; Armitage et al., 2007; Sarcevičius and Taraškevičius, 2015). We here conduct our own baseline studies of brick geochemistry to discover what information our bricks may record.

Our approach was to core the bricks with a diamond coring tool and grind a fresh flat surface on the core plug, revealing the very heterogeneous substances comprising the brick. Our first pass through the 8 bricks extracted cores of diameter about 19mm. These were

examined and the ground surface was analyzed chemically along LAICPMS burn tracks about 1 cm long and 150 microns wide. Petrographic examination of the ground core surfaces revealed a large variety of recognizable components: reconstituted clay, sand grains of several types, rock fragments, clinker, and coal waste. A laser track across the exposed surface ablated the target material as plasma which was fed into a quadrupole mass spectrometer for elemental abundance analysis. Our tracks were several mm to a cm in length and about 150 microns wide, and sampled the heterogeneous substances comprising the bricks. A time series of plasmas fed into the mass spectrometer was displayed as a time series of detected elemental signal intensities. Backgrounds and signals were picked from the time series and processed to give elemental abundances. NIST glasses 610 and 612 were used as standards to calibrate mass intensities in the spectrometer into actual abundances. Examination of the first pass 19mm cores revealed systematic similarities and differences between the bricks, so a second pass analysis (brick\*) was undertaken to judge how reproducible were the results. The scale of heterogeneity seen in the time series of the first pass was sub-mm, so the samples of the second pass were taken with a 6mm coring tool and laser tracks of only 3-4 mm long by 150 microns wide were considered sufficiently long to recapture the significant variations. These smaller cores were taken from the other end of the same bricks sampled in the first pass. There were only 7 bricks sampled in the second round (Brick\*) as degraded brick DATR was not sufficiently coherent to be cored on the smaller scale. Pictures of the laser tracks showing sample surface heterogeneity are in the appendix.

### **Samples studied with brief description**

**SC** Brick ends sawn from material from Ft. Sint Kruis, supplied by W.A. Taylor. Well indurated yellow brick of smaller size than common modern brick.

**DUCY** Entire tablet of degraded Dutch yellow brick from Curacao supplied by Dr. Charles Gehring of the New Netherlands Institute of the New York State Library. Material known to be Dutch in origin and of possible contemporary interest for the construction of Ft. Sint Kruis. It serves as a comparator of Dutch-sourced brick of the 1630s, for comparison to the SC sample of uncertain provenance. Both bricks are of similar dimensions, being smaller and thinner than modern brick. The DUCY brick is quite badly degraded from ~400 years of marine exposure and erosion. [See Figure 1.] Dr. Gehring provided a preliminary report of X-ray fluorescence analysis (background information in appendix) indicating high levels of Ca and Cl in this brick, subsequently confirmed in our study.

**HVR** Red brick with ROSE imprint from the Roseton, NY brickyard of Rose Bros., supplied by D. Walker as a Hudson River brick industry comparison sample.

**HVCX** Red brick with XXX imprint from the Catskill, NY brickyard of George W. Washburn Co., supplied by D. Walker as a Hudson River brick industry comparison sample.

**DATY** Thin yellow Danish-sourced brick supplied by W.A. Taylor for comparison purpose.

**DATR** Thin red Danish-sourced brick supplied by W.A. Taylor for comparison purpose.

**DAFY** Fat yellow Danish-sourced brick supplied by W.A. Taylor for comparison purpose.

**DAFR** Fat red Danish sourced brick supplied by W.A. Taylor for comparison purpose.

### **LAICPMS analytical procedure details**

Sample analyses were performed using an ESI/ New Wave UP-193nm Excimer Laser Ablation system in conjunction with a VG PQ Excell quadrupole ICPMS. Samples were ablated in He filled sample cell with flow rate about 1.5 L/min. The ablated material was then carried by a He and Ar gas mixture into the ICPMS torch where the material was ionized. The gas at the torch was roughly 50:50::He:Ar at a flow rate of about 3 L/min.

We used a large spot size, 150 $\mu$ m in diameter, in an attempt to see a more homogenized signal. We lasered lines across the samples to see any variability across a single sample. Laser power used was approximately 1.2 GW/cm<sup>2</sup> with a frequency of 10Hz and a tracking speed of 5 $\mu$ m/s. Each nuclide was monitored with a 10 ms dwell time and detected in pulse mode up to 10<sup>6</sup> counts/s, above which the detector automatically switched to analog voltage mode. Such high counting rates were only encountered for major elements Na, Mg, Si, and Fe in this study. Because lasering was purely surficial, the sample's surface needed to be cleaned. We cleaned the surface of any contaminants by using a much softer beam, about 20% of the laser intensity moving at a speed of 100  $\mu$ m/second with a slightly larger spot size of 175  $\mu$ m. This removes a very thin layer of the surface, leaving fresh sample to ablate and analyze. A background for each sample was measured for 60 seconds on the ICPMS without any laser ablation occurring. Two standard glasses, NIST 610 and 612, were laser ablated to help quantify the sample concentrations. These standards are Ca-Al-Na-Silicate glasses doped with approximately 400ppm (610) and 40ppm (612) of most of the other elements measured. Since the NIST glasses are so concentrated, a smaller spot size of 50 $\mu$ m was used with a pre-ablation spot-cleaning size of 65 $\mu$ m.

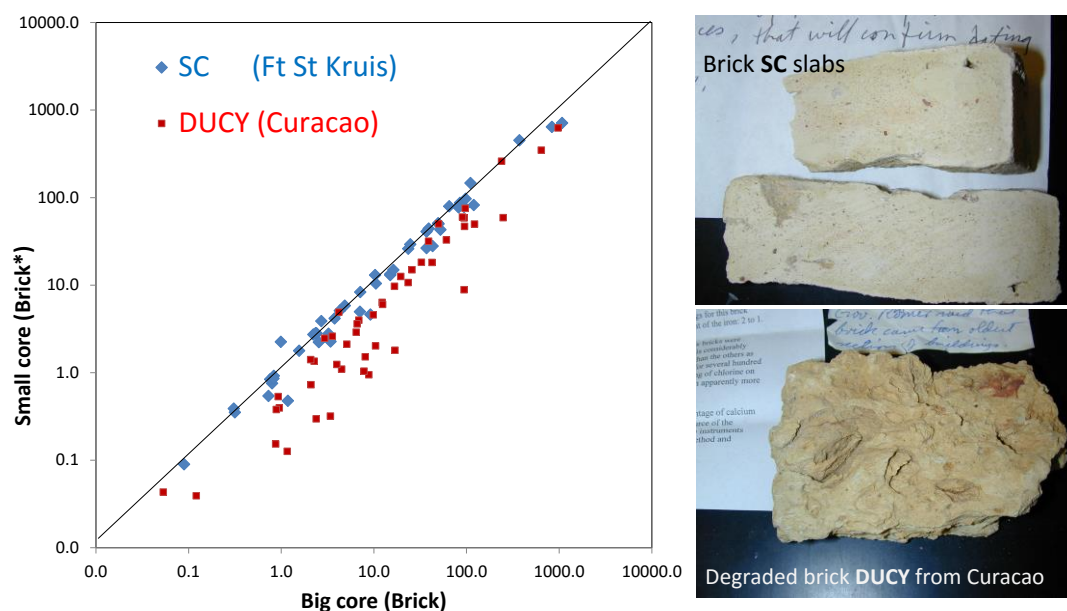
Data reduction was done by first looking at the time series for the samples and standards graphically. An average intensity was calculated for the time-resolved ablation (TRA) segments that best represent the sample or standard analysis and a TRA segment of its corresponding background. The average background intensity was then subtracted from the average analysis intensity. This removes any background caused by noise or interfering gas complexes. Since bricks and silicate glasses do not ablate equally and different spot sizes were used, some element must be used as an internal standard to correct for these variations. This element must have a known concentration. We chose Si as our internal standard. Each background corrected intensity for a given sample or standard was then divided by its background corrected intensity of Si and then multiplied by the Si concentration of that sample

(used a generic 50%) or standard (exactly known). Sample concentrations were then calculated by using a standard curve consisting of NIST 610, NIST 612, and forcing the line through the origin. Since there is no Hg202 in the NIST glasses we used the calibration slope of Pb208 and corrected it for the difference in isotopic composition of Pb and Hg. This gives us an approximate value (uncertainty  $\sim \pm 20\%$ ) for Hg because the difference in ionization potentials between the two elements is not accounted for.

### LAICPMS results

Table 1 gives the results of the 15 LAICPMS analyses of the 8 bricks. Those 7 with \* designation (Brick\*) are analyses from the smaller cores of the second analysis group which have a more comprehensive analysis, including Cl and Mo, than the (Brick) analysis of the larger core from the opposite end of the same brick. A graphic representation of the reproducibility of the technique is given in Figure 1 by plotting the values determined for all elements, with the value for (Brick) as the abscissa and (Brick\*) for the ordinate. If there were perfect agreement between the duplicate analyses, they would fall along a 1:1 line. The duplicate analyses of the two bricks from the Caribbean, SC and DUCY, are shown in Figure 1.

**Figure 1** Comparison of duplicate samplings for Caribbean bricks with their pictures showing contrasting states of degradation by weathering.



**Table 1** Analytical results

Atomic Mass #	Element (by wt.) ppm or %	SC	SC*	DUCY	DUCY*	HVR	HVR*	HVCX	HVCX*	DAFY	DAFY*	DATY	DATY*	DAFR	DAFR*	DATR	Upper Crust Rudnick & Gao
		Caribbean				Hudson Valley				Danish							
7	Li	37	41	39	32	42	35	57	43	25	26	47	40	43	50	60	24
9	Be	2.4	2.8	2.3	1.4	1.5	1.5	2.3	2.1	1.8	1.5	2.1	1.9	2.5	2.2	2.6	2.1
11	B	65	80	94	59	79	52	76	66	74	49	90	70	68	68	85	18
23	Na2O%	2.6	2.2	4.5	1.1	2.0	1.4	1.0	0.7	1.8	3.4	7.2	1.6	0.8	0.8	1.0	3.27
25	MgO%	3.8	4.2	2.9	2.5	2.2	2.6	1.8	1.8	1.8	2.3	2.8	2.7	1.3	1.5	2.8	2.48
29	SiO2%	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	50.0	66.6
31	P	1075	711	641	349	864	558	625	358	863	454	753	369	817	571	999	645
35	Cl		1114		1275		487		368		2544		916		438		370
43	CaO%	37.0	26.6	19.6	12.6	11.4	4.5	2.5	2.0	24.8	20.0	20.5	9.9	1.3	1.0	30.5	3.9
45	Sc	15.1	13.4	12.3	6.4	10.2	8.4	14.0	11.2	9.7	6.7	9.7	6.5	13.2	8.3	15.7	14
47	TiO2%	0.8	0.8	1.0	0.4	0.9	0.7	0.8	0.7	0.6	0.5	0.6	0.6	1.0	0.5	0.9	0.64
51	V	85	87	122	50	93	61	98	74	58	49	96	52	110	81	124	97
52	Cr	94	78	91	60	39	47	67	59	54	45	109	52	47	97	83	92
55	MnO%	0.09	0.09	0.05	0.04	0.12	0.11	0.08	0.09	0.08	0.09	0.04	0.03	0.07	0.04	0.08	0.10
57	FeO%	9.2	4.6	5.1	2.1	6.2	3.2	7.7	3.7	5.3	2.7	6.9	3.0	6.6	3.3	8.7	5.0
59	Co	15	13	12	6	14	12	15	12	11	14	7.4	4.8	10	6.3	14	17
60	Ni	52	43	42	18	45	26	56	32	42	31	39	19	54	21	47	47
65	Cu	43	28	23	11	81	31	42	25	51	19	34	7.4	18	10	41	28
66	Zn	120	82	95	47	117	148	116	72	81	50	85	42	82	55	114	67
85	Rb	82	77	97	75	81	77	142	132	90	47	87	77	121	97	122	84
86	Sr	835	643	973	626	293	147	112	127	678	441	494	266	97	90	656	320
89	Y	24	26	94	9	46	26	17	27	27	15	12	11	21	15	27	21
90	Zr	111	147	248	59	235	308	75	135	137	85	39	60	95	192	194	193
93	Nb	16	15	17	10	19	13	14	14	12	8	11	11	17	10	17	12
98	Mo		0.34		0.17		0.22		0.23		0.39		0.20		0.30		1.1
111	Cd	1.2	0.48	0.12	0.04	0.07	0.53	0.08	0.13	0.75	0.47	0.37	0.07	0.04	0.06	0.58	0.09
118	Sn	3.2	2.8	3.6	2.6	2.8	1.9	3.3	2.6	2.3	2.0	2.9	1.9	3.0	4.3	2.9	2.1
133	Cs	7.1	5.0	6.9	4.0	3.1	2.8	6.1	5.2	6.0	4.4	5.6	5.0	9.0	7.8	5.1	4.9
138	Ba	373	451	240	260	299	319	432	597	413	439	250	189	237	230	440	628
139	La	49	51	33	18	37	33	34	41	98	52	24	22	25	27	45	31
140	Ce	98	97	61	33	79	64	79	77	213	105	45	43	52	54	89	63
141	Pr	11	10	6.6	3.6	9.0	7.1	7.8	8.0	22	11	4.7	4.9	5.5	5.9	9.6	7.1
145	Nd	39	44	26	15	37	31	30	35	82	38	18	19	22	23	37	27
147	Sm	7.1	8.3	6.4	2.9	8.1	6.4	5.8	6.9	15	6.5	3.7	3.7	4.5	4.7	6.9	4.7
153	Eu	1.6	1.8	2.1	0.7	1.9	1.4	1.2	1.4	1.7	1.3	1.0	0.86	1.0	1.0	1.5	1.0
160	Gd	4.9	5.8	10.4	2.0	7.2	5.0	3.8	5.3	9.3	4.5	2.4	2.6	3.8	3.6	5.0	4.0
159	Tb	0.8	0.8	2.4	0.30	1.2	0.77	0.57	0.77	1.4	0.70	0.38	0.42	0.62	0.56	0.81	0.70
163	Dy	4.3	5.1	16.9	1.8	7.8	5.0	3.2	4.9	6.6	3.7	2.1	2.4	3.7	3.2	4.9	3.9
165	Ho	0.8	0.9	3.4	0.32	1.7	0.91	0.63	0.89	1.1	0.69	0.39	0.47	0.75	0.64	1.0	0.83
166	Er	2.2	2.7	8.8	0.95	4.7	2.7	1.7	2.7	2.5	1.7	1.0	1.2	2.0	1.7	2.6	2.3
169	Tm	0.32	0.35	1.2	0.13	0.65	0.35	0.25	0.35	0.32	0.23	0.14	0.18	0.29	0.26	0.38	0.30
172	Yb	2.4	2.8	7.8	1.0	4.6	2.8	1.8	2.7	2.3	1.7	1.1	1.3	2.1	1.9	2.8	2.0
175	Lu	0.31	0.39	0.87	0.15	0.6	0.4	0.25	0.38	0.31	0.25	0.14	0.19	0.29	0.29	0.38	0.31
178	Hf	2.7	3.9	8.1	1.5	6.2	7.0	1.9	3.3	3.8	2.7	1.0	1.9	2.7	5.5	4.8	5.3
181	Ta	0.82	0.86	0.93	0.53	1.0	0.70	0.77	0.79	0.67	0.49	0.52	0.73	1.1	0.78	0.86	0.90
182	W	1.0	2.2	2.1	1.4	2.5	0.94	1.2	1.1	1.1	2.2	1.4	1.5	2.3	1.3	2.0	1.9
202	Hg	0.7	0.5	0.9	0.4	0.6	0.70	0.47	0.66	0.47	0.49	0.41	0.49	0.70	0.92	0.48	0.05
208	Pb	25	29	4.2	4.9	19	17	24	17	22	23	13	7.7	24	37	24	17
232	Th	10	13	9.9	4.6	7.9	8.9	9.3	10	28	14	5.3	6.5	8.4	10	11	11
238	U	3.4	2.3	4.0	1.2	2.9	2.1	2.5	1.9	5.6	2.8	1.4	1.8	2.0	2.7	3.2	2.7

SC is representative of the most reproducible results for small and large cores that we obtained. DUCY is atypical and is easily the least reproducible result obtained among our 7 sets of duplicate analyses. There is very respectable conformity to 1:1 for the blue SC array, indicating both good reproducibility of analysis and good correspondence between the compositions at different ends of sample SC (unless there is some implausible Maxwellian conspiracy between biasing errors and real differences between cores of the same brick!). In contrast there is a clear bias to the DUCY analyses with the DUCY\* values being systematically lower than those for DUCY. There is also about an order of magnitude scatter to the results in DUCY compared to the better than a factor of 2 agreement within the SC results. The systematic  $\times 2$  depletion of DUCY\* could be analytical, for instance in the SiO<sub>2</sub> content that was used for normalization and assumed to be a generic 50%. However SiO<sub>2</sub> cannot be in error by

the factor of 2 needed, and we will show below that there clearly are real and detailed differences between the opposite ends of DUCY. This difference between ends is also clear from the larger scatter in the red DUCY array.

**Figure 2** Analysis of 50 elements in small cores given in ppm by weight.

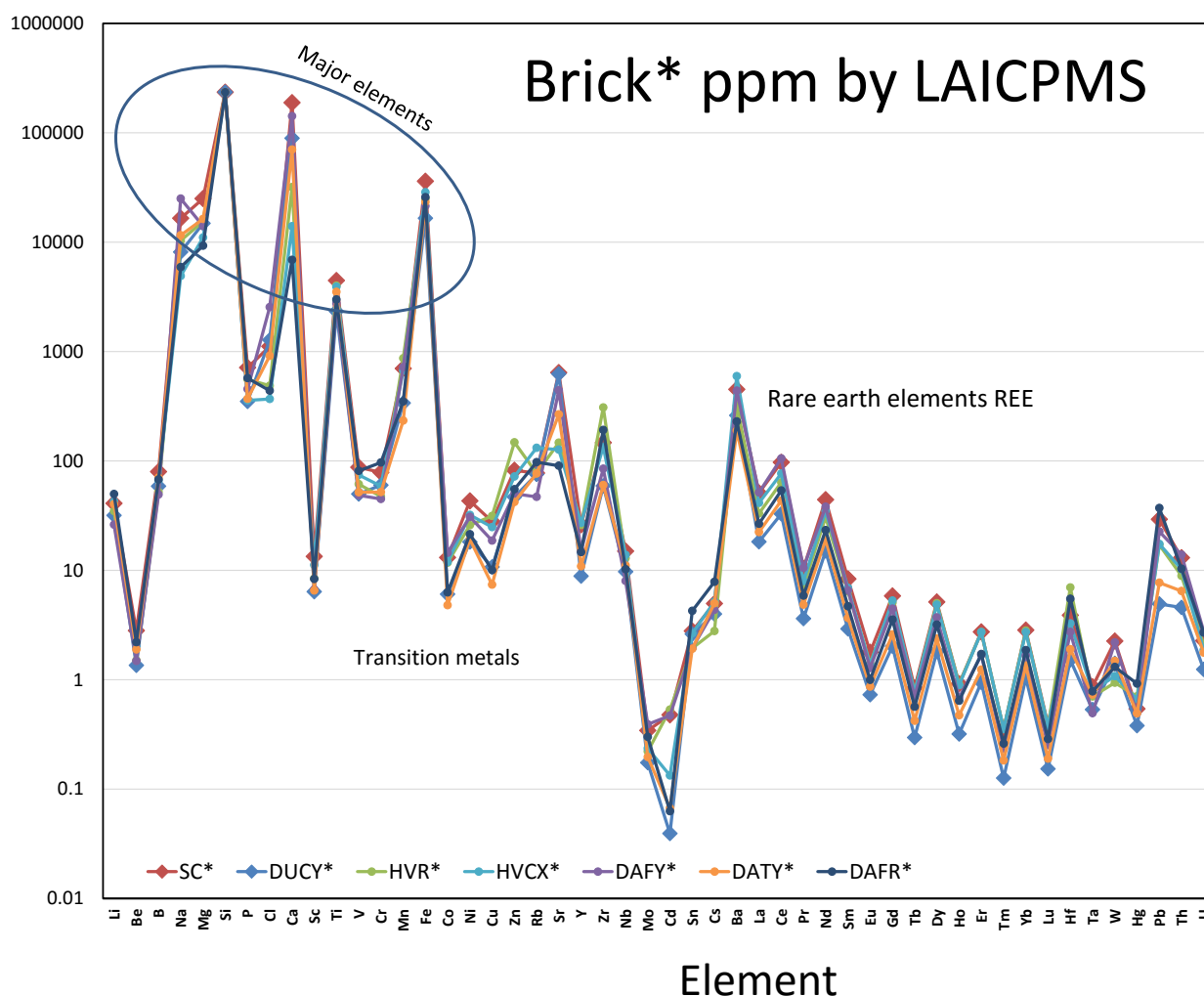


Figure 2 gives the results of 50-element analyses for 7 bricks from the more comprehensive analyses of the second pass (brick\*) on the smaller core samples. The elements on the abscissa are given in increasing atomic number, with the ordinate being the value of the concentration of that element by weight in parts per million (ppm). The elemental abundances cover a range of values approaching 7 orders of magnitude. There is a general trend of decreasing elemental abundances from the major elements at low atomic number, through the transition metals, to the rare earth elements, to the heavy metals ending with U. Exceptions to this general trend come from low abundances for the very light trio Li-Be-B, from Sc, and from moderately heavy metals Mo and Cd, all of which are conspicuously under-abundant compared to the general decrease in abundance with atomic number. There is also a conspicuous zig-zag

modulation to the abundances following the Oddo-Harkins even-odd atomic number rule reflecting nucleosynthesis effects on elemental abundances. This modulation is especially noticeable on the heavy side of the Cd-depletion valley, for instance among the rare earths La to Lu.

What is especially striking in Figure 2 is that all 7 bricks, from both sides of the Atlantic, with manufacturing ages from the 17<sup>th</sup> to the 20<sup>th</sup> centuries, and with strongly contrasting states of degradation by weathering, all show very similar patterns of elemental abundances. The zigs and zags are all over the map covering almost 7 orders of magnitude variation, but the 7 bricks all follow pretty much the same script, whatever that script is. **It is clear that this broad survey mode of geochemical analysis is unlikely to provide easy discriminants of provenance, manufacture, or degradation history because these bricks all present as one at this scale.** A much finer examination of specific features will be needed.

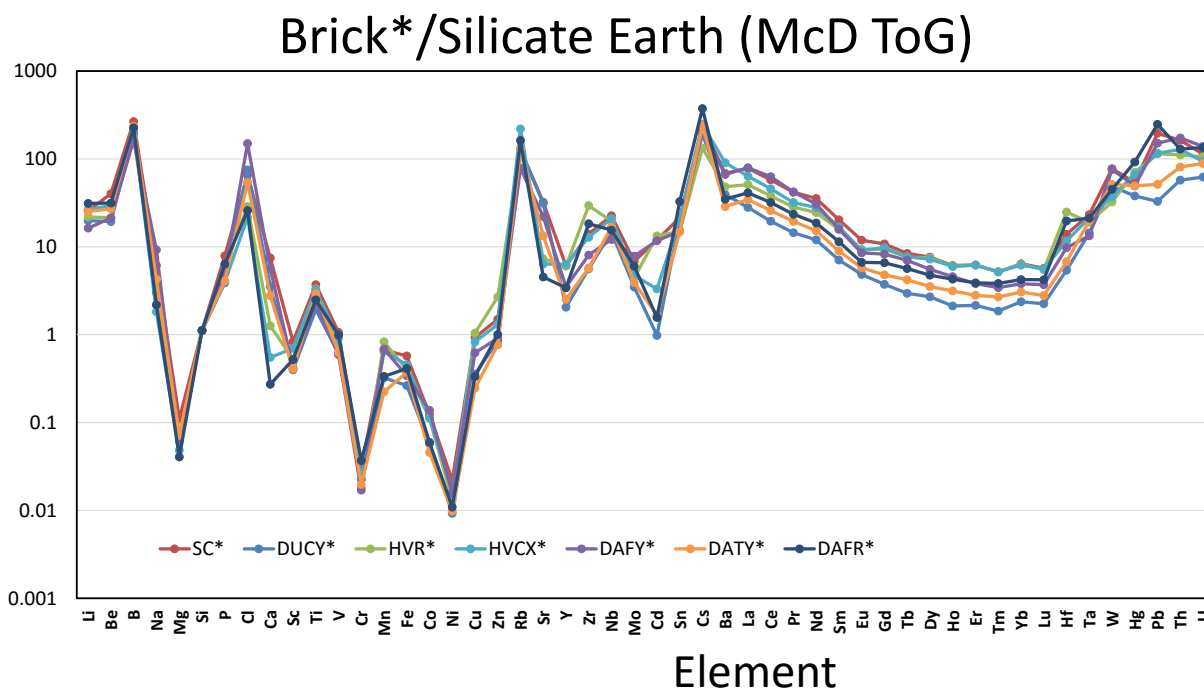
Much of the eye-catching variation of overall decrease with atomic number and Oddo-Harkins even-odd modulation can be understood as nucleosynthetic effects, which are somewhat distracting from our purpose of extracting any potential information about the provenance of the source materials, the manufacture, or the degradation of the bricks. The usual procedure used to remove those distractions is to normalize to other reference materials that also contain those same nucleosynthetic signatures. The photosphere of the sun and some classes of chondritic meteorites have similar enough composition in terms of the relative abundances of the elements we measure (excluding H and He) to serve as a proxy for the elemental abundances of planetary building materials forged during nucleosynthesis before the solar system's birth. The bulk silicate Earth is thought to be roughly 'chondritic', so we show in Figure 3 the information in Figure 2 normalized by dividing by the elemental abundances given by McDonough (2003) for the silicate Earth estimated by reference to chondritic meteorites and other possible measures of the Earth's composition. This normalization is quite successful at removing the overall slope of the plot and in removing the distracting Oddo-Harkins even-odd modulation. The overall variation is now reduced by a couple orders of magnitude through the chondritic silicate Earth normalization. But considerable structure remains in the normalized data, which still shows a range of more than 4 orders of magnitude, even though individual elements all show less than an order of magnitude variation among the 7 bricks. The average of all the normalized values in Figure 3 is not close to one, from which we learn that bricks are not like the silicate Earth in their elemental abundances. This should not be a surprise. Bricks are highly differentiated geochemically from bulk planetary compositions.

The very distinct structure seen in Figure 3 includes significant depletions in Mg (instead of Be), Cr, and Ni and significant excesses in B, Cl, Rb, Ba, and Pb compared to the silicate Earth. The rare earth elements (REE), especially the light REE (LREE), are all coherently enriched in all the bricks compared to a chondritic Earth. What is this structure's meaning? To help answer this question, in Figure 4 we present the data of Figure 2, now normalized to the average abundances in the upper crust of the Earth given by Rudnick and Gao (2003). The large

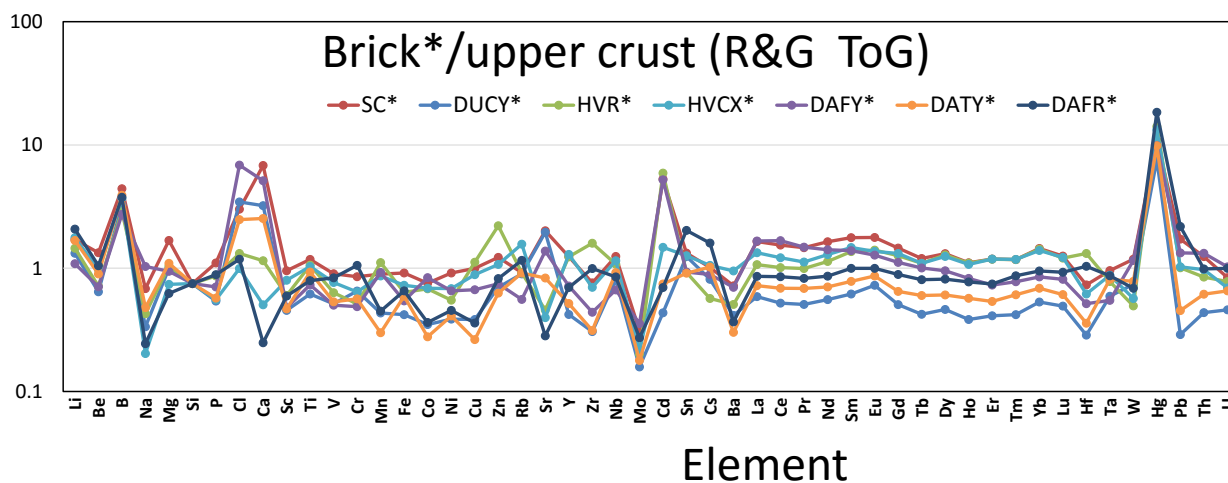


variations of Figure 2 and 3 collapse to a narrow band within an order of magnitude of the value 1. The order brought to the geochemical patterns of the bricks by normalization by average continental crust suggests we now have an appropriate baseline from which to hunt for smaller signals that may be informative about bricks rather than the gross geochemistry of their feedstocks in the upper continental crust. We can now use this normalization to see through the geochemical complexity of nucleosynthesis, Earth differentiation, and upper crust formation.

**Figure 3** *Small cores normalized to the silicate Earth of McDonough (2003)*



**Figure 4** *Small cores normalized to upper crust of Rudnick and Gao (2003)*



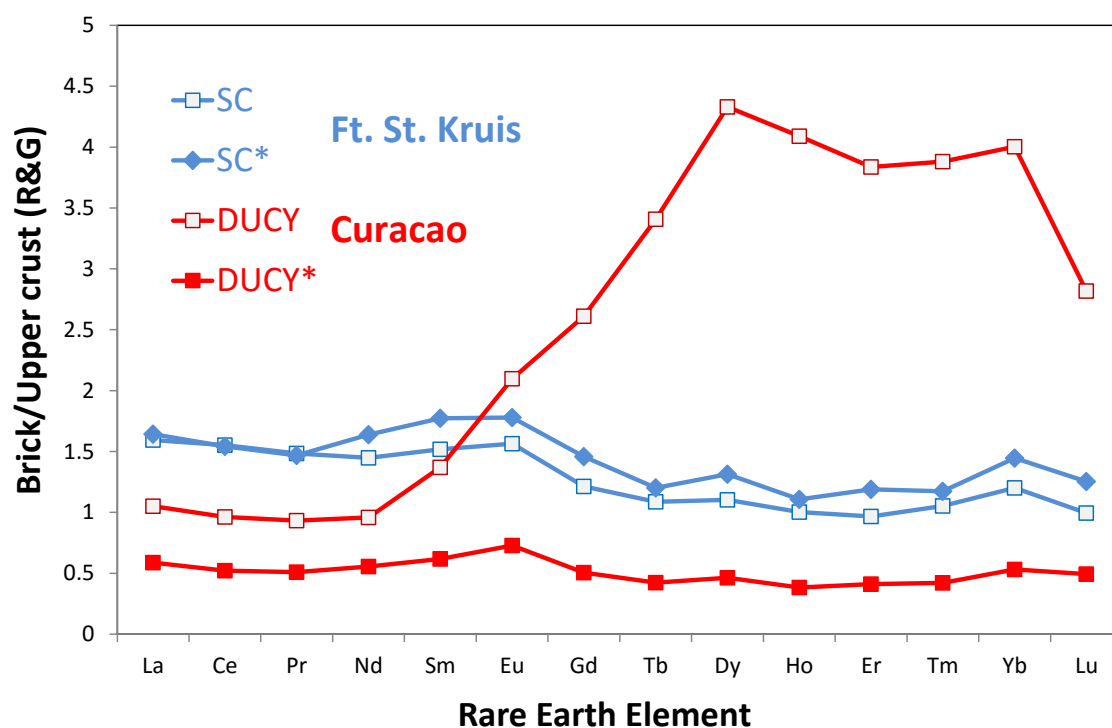
Clearly bricks look like upper crust of the Earth -- bricks from a wide range of places, ages of manufacture, and states of degradation. Elemental exceptions to this are Hg and perhaps Cd. The Hg values have little scatter and are all high. This could be a result of using the calibration slope of Pb208 instead of that for Hg202 because the NIST glasses have no Hg. We estimate that the Pb-for-Hg approximation might raise uncertainty to the 20% level rather than the 1000% level needed to explain the factor of 10 excess of Hg. It is also possible that the upper crustal value for Hg is poorly known. Rudnick and Gao report order of magnitude discrepancies between various sources. Thus we do not feel that Hg necessarily overturns our first order conclusions that bricks strongly resemble upper continental crust in their elemental abundances. This conclusion is satisfying. Bricks are made from clay and sand which are derived from the weathering of the upper continental crust. What is perhaps surprising is that the chain of processes in the formation of bricks includes the decay of upper crust by the weathering process which is chemical in part. It includes the blending process of sand, clay, and exotic aggregates (e.g. coal waste in 19<sup>th</sup> century Hudson valley bricks, lime in some of the older bricks) which are chemically unrelated to each other. It would not be impossible that the firing process of consolidating the blend of raw materials could leave its own chemical signature. The weathering process by which some of the bricks suffered degradation could also be in part chemical. And yet it is remarkable that this combination of processes should so faithfully preserve the upper crustal signature for so many elements seen in Figure 4. The normalization of brick chemistry to upper continental crust provides a backdrop against which process and provenance signals can be more readily recognized

There are subtle, but recognizable, signatures of some of the process steps. For instance the variations of Ca undoubtedly reflect the variable addition during brick manufacture of lime to the mix to better bond the sand grains to the reconstituting clay body during thermal consolidation. High lime contents are also effective at suppressing the red color of Fe oxides, and indeed the red bricks\* in Figure 4 are low lime, whereas the yellow bricks\* are high lime. Likewise the excess-Cl bricks may reflect weathering in a marine environment for ~400 years. The bricks from the 19<sup>th</sup> century, fresh water Hudson River valley are not enriched in Cl (both less than 500 ppm), whereas the Curacao and Fort Sint Kruis bricks, which are known to have been exposed in a marine weathering environment, are Cl enriched (1100-1300 ppm). It is also possible that their source clays may have been pugged with sea water. This Cl enrichment was previously known from Dr. Gehring's XRF report on the Curacao brick DUCY (appendix), as was its high lime content. These glimpses into process provided by 2 specific elements, Ca and Cl, are obscured by the much larger body of geochemical information from 46 elements (Hg and Cd are rogue) that consistently says "UPPER CRUST" without regard to source material provenance, manufacture, or subsequent degradations. While an interesting conclusion in its own right, it does little to advance our original objective of finding the source of particular bricks.

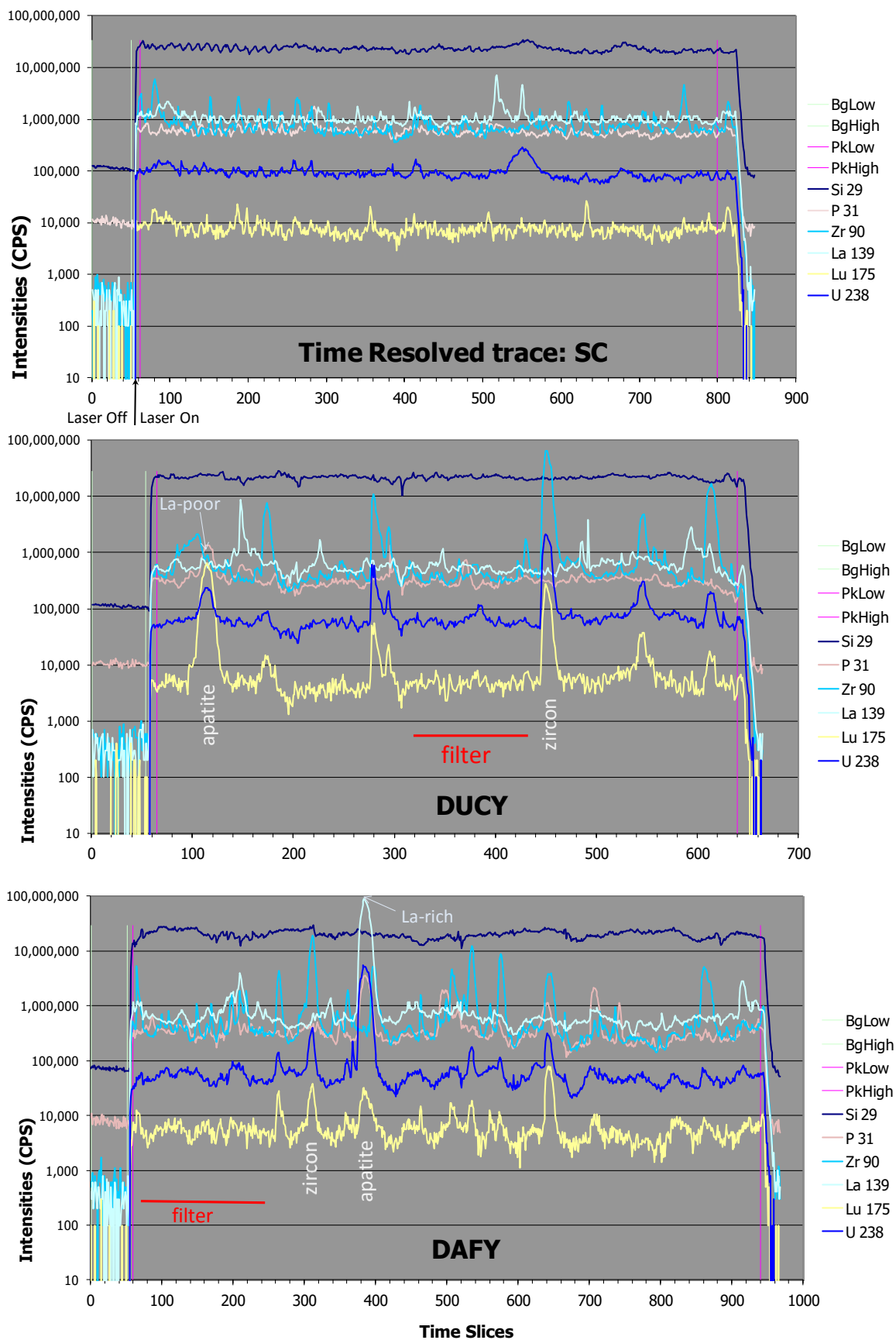
## Spatial Complications

Our first order, broad-brush, bulk geochemistry conclusion that “bricks = upper crust”, develops some wrinkles on a finer spatial scale. We made two separate groups of analyses of the bricks from different sized cores taken from opposite ends of the same bricks. We have initially examined the data from the pass through the 7 smaller (brick\*) cores because they included elements Mo and Cl that were not analyzed in the pass through the 8 larger (brick) cores. We now additionally examine the (brick) data from the larger cores to check the reproducibility of the results for any particular brick. Does one get the same answer from different ends of the same brick? Figure 1 and the typical results for SC vs. SC\* suggest that the answer is usually, approximately yes. SC closely conforms to the 1:1 line in Figure 1. This ‘yes’ answer is consistent with the lack of much brick to brick variation in the (brick\*) sampling of small cores in Figure 2 that lead eventually to our broad conclusion in Figure 4 that “bricks = upper crust”. But there are still some interesting within-brick variations, in addition to the process-related variations in Ca and Cl. These additional variations within one brick were seen for example, in the large scatter shown by DUCY in Figure 1 for all the elements. Clearly the opposite ends of DUCY are measurably different from each other, and also from its closest neighbor SC-SC\*.

**Figure 5** REE in Caribbean bricks sampled twice. Open symbols, big cores; filled symbols, small cores\*



**Figure 6** Time/position traces along laser tracks for SC, DUCY, and DAFY large cores



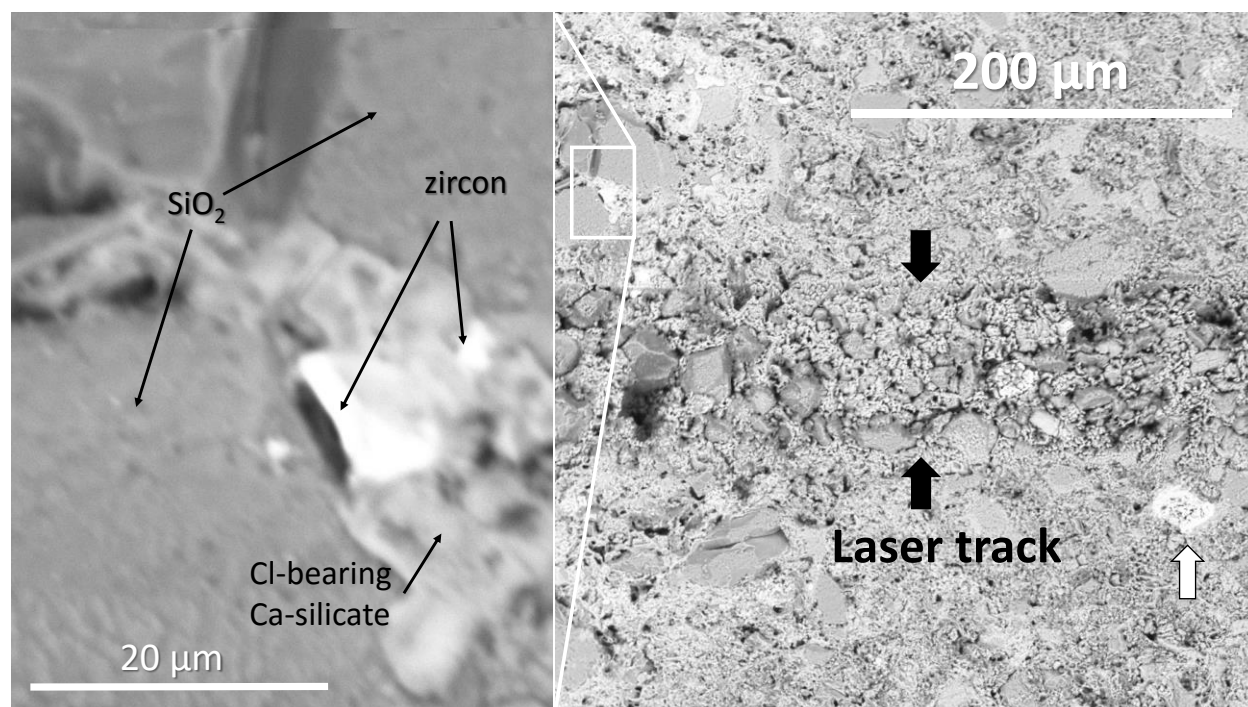
Within all elements measured, the REE are a particularly coherent and well-behaved suite. We concentrate our further examination on these elements for the present discussion which continues the comparative study of the two Caribbean samples, SC and DUCY.

Consistent with Figure 1 and its 1:1 slope for SC-SC\*, Figure 5 shows that the REE are highly reproducible in the two samplings of brick SC from Fort Sint Kruis. The blue curves are not distinguishable within analytical uncertainty. The LREE are very marginally enriched compared to the HREE on an upper crust-normalized basis. In contrast to SC and most other bricks, the two samplings of the badly degraded Curacao brick are utterly different from each other. DUCY\* small core that we examined previously is slightly depleted in REE but unfractionated relative to upper crust, like most of the other bricks. DUCY\* filled red squares have a nearly flat profile at a slightly depleted value near  $\frac{1}{2}$ . The DUCY large cores however show strong fractionation compared to upper crust, with marked enrichment in the HREE of about a factor of 4 compared to upper crust. Why is this?

The differences between DUCY and DUCY\* are best appreciated in the context of the sameness of SC and SC\*. There are some fundamental differences in the spatial distribution of elements in the different Caribbean samples. Figure 6 shows the time-resolved ablation traces for selected elements for the two Caribbean samples SC and DUCY, as well as the trace for one Danish sample DAFY. DAFY differs significantly from DAFY\*, although not as much as DUCY differs from DUCY\*. The abscissa of these plots, the time slice values, correspond to position in the burn line as the laser tracks across the sample surface once the laser has been turned on. The initial data collection from the mass spectrometer, with the laser off and the sample stationary, measures instrument background. The traces for SC shows fairly uniform intensities of the signals for Si, P, Zr, La, Lu, and U, although not as uniform as for the NIST glass standards. The fairly uniform signal is generated from a very heterogeneous target with sand-sized grains clearly visible along the traverse, in this and in all the other samples. Small rises and falls of a factor of 2 in intensity with the occasional order of magnitude blip in some elements like Zr or U are characteristic of this mode of sampling on the scale of the 150 micron wide laser track. There is little difficulty in producing representative averages of the signals that faithfully represent the bulk of the fairly well behaved signal. This well-behaved signal with time/position is not characteristic of DUCY or DAFY. Both show spikes, even plateau, of elevated signal for the elements shown, except for Si. The bumps are blasts of signal intensity of up to an increase of a factor of 100 on an otherwise smooth background that resembles closely the signals from SC. These injections of extra signal imply that the differences between DUCY and DUCY\* are a result of this extra signal in DUCY, not signal deficiency in DUCY\*. This is clear from the details of the bumps and their occurrence. DUCY is shown to be HREE enriched in Figure 5. The bumps in DUCY for Lu are very large, but they are almost nonexistent for La, so the bumps are clearly the reason for the DUCY HREE enrichment in Figure 5. Of the half dozen significant bumps in the DUCY trace for Lu, there is an exactly corresponding bump for Zr and U for the last five. Zircon can be U-rich and HREE-rich, suggesting these 5 bumps may be small individual zircons caught within the laser beam. The unperturbed traces for the major elements suggest that the

individual zircon crystals are small compared to the beam size of 150 microns. Indeed, a cursory SEM examination of DUCY with BSE imaging in Figure 7 shows that there is a small population of zircons as well as rutile, ilmenite, and apatite among the mineralogically recognizable collection of granular ingredients. Figure 7 shows one very large ( $\sim 10\ \mu\text{m}$ ) zircon and two more representative smaller ones ( $1\text{--}2\ \mu\text{m}$ ) wedged between temper grains of  $\text{SiO}_2$ . Grains of these sizes would be completely ablated in the laser track which is more than 10 times wider than the particle dimensions, consistent with finding no zircons in the track itself during the SEM exam. The zircon signature of high Zr is seen and recorded, however, in the time-resolved trace of plasmas in the mass spectrum.

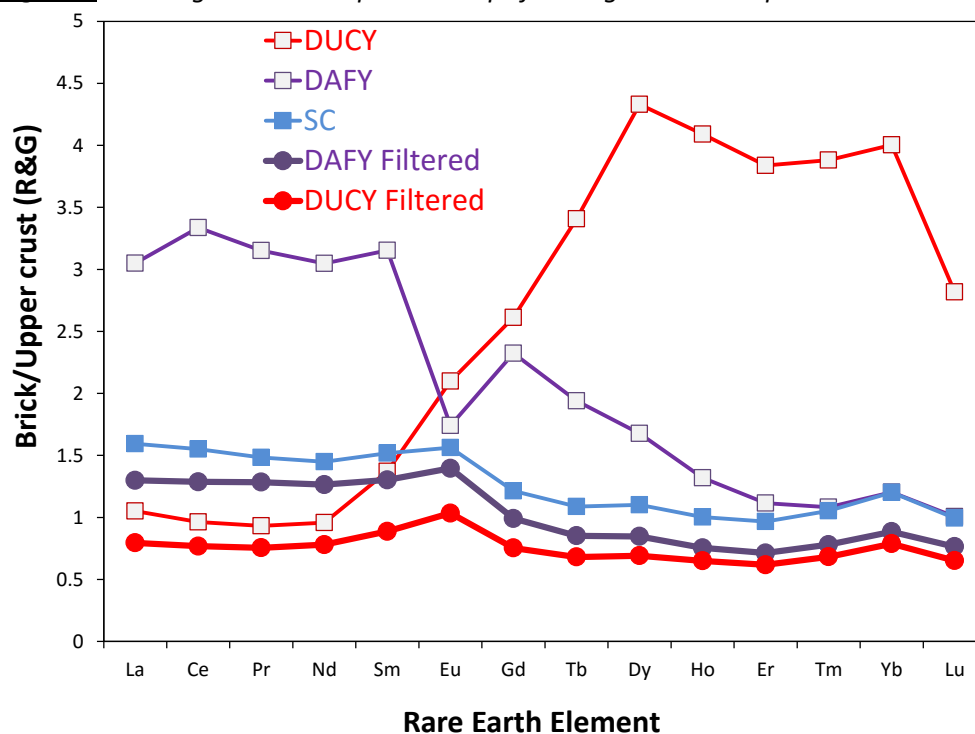
**Figure 7** SEM/BSE images of DUCY. Laser track, delimited by black arrows, runs horizontally across right image. Granular heterogeneity is in full display. Enlargement of zircons in left image close to, but outside, the laser track. This lime-rich brick has complex Cl-bearing Ca-silicates as matrix binder for some of the temper grains. It is unknown whether the Cl-bearing nature of these silicates is a result of marine weathering or is original in the firing reactions. The bright diamond-shaped grain indicated by the white arrow in the right image has the shape and Ti-rich, Fe-poor signature of rutile, however it is also Ca and chlorine bearing, unlike the  $\text{SiO}_2$  grains of the temper, and its geometry is reactive. It is difficult to imagine rutile suffering this sort of degenerative reaction from marine exposure. Perhaps the brick-making before the firing process may have seen some chloride contamination or perhaps pugging with sea water that was reactively consolidated with the brick's firing. This is yet another sort of petrographic idiosyncrasy that may prove useful in distinguishing one brick source from another.



The correspondence for the first bump at time slice 112 for DUCY in Figure 6 is not with Zr, but with P and U, suggesting that a phosphate apatite grain is the source of the first extra

signal, not zircon. The ‘noise’ that makes the differences between DUCY and DUCY\* is apparently a variable population of microscopic mineral grains of REE-rich accessory phases like zircon and apatite. When the beam samples these nuggets, the elemental patterns are perturbed. DAFY shows similar nugget-related bumps and spikes and has a fractionated average value showing LREE enrichment. The largest bump near time slice 380 in DAFY has La>Lu with much U and P but little Zr, suggesting that apatite is the culprit, not zircon. The pattern of La>Lu in this huge bump is consistent with the LREE enrichment shown by pattern DAFY in Figure 8. If we were to filter out the bumps introduced by zircon and apatite grains within some bricks, one might expect a return to the “bricks = upper crust” baseline. We have done this exercise by including in the analytical averages only the signals in Figure 6 within the window of the red ‘filter’ bar. These filtered results are given in Figure 8 with the unfiltered results. Filtering does return bricks DUCY and DAFY to the pattern of SC with remarkable fidelity. SC is an archetype for the “bricks = upper crust” story line. SC, having no bumps, requires no filtering. SC, filtered DUCY, and filtered DAFY all converge on flat patterns with normalized values of  $1(\pm\frac{1}{2})$  in Figure 8. This sameness of base signal from Dutch, Danish, Caribbean (and Hudson Valley) bricks presents a challenge for assessing provenance. The signals of the accessory particles, their presence or absence, their identity, their abundances, and trace element signatures (which are quite variable), potentially have more provenance information than the baseline. **The accessory particles represent a better target for study.**

**Figure 8** Filtering zircon and apatite bumps from signals returns patterns to those like SC.



The heterogeneity of samples, down to and including laser beam sampling scale, is both a challenge and an opportunity. The challenges of population heterogeneity have been extensively examined by Gilbert et al. (1993). The more heterogeneous the population, the more work required to properly characterize it. Cluster analysis, to recognize distinct populations of objects, requires an adequate sampling to properly delineate the separateness or sameness of populations. And principal component analysis likewise requires adequate sampling to delineate any important multicomponent covariations. Heterogeneity is implicit in clusters and components, so it is an exercise in sampling scale to sort noise from signal. On too coarse a scale bricks become indistinguishable from upper crust. The crustal signal has its characteristic covariation of elements, and it is best to remove that signal shared by all our bricks by crustal chemistry normalization. On too fine a scale bricks become granular agglomerations of mineral particles. And yet those mineral particles have some prospect of being useful signal carriers of source materials. That opportunity is lost if sampling is for bulk chemistry on a scale too much larger than the particles. Now that we have identified individual mineral types as the carriers of the distinctive signals which overprint the crustal background signal, the next stage of research might profitably focus on characterizing such particles. This has been done for the different bricks of old Toledo by López-Arca et al. (2003) using garnet as a tracer of different clay deposits upstream and downstream from garnet-bearing bedrocks in the Toledo area.

### **Implications**

Individual bricks are revealed to be chemically heterogeneous on several scales, including that of the laser beam track. These heterogeneities make it difficult to find easy, definite conclusions about brick provenance based on a single chemical analysis, because different ends of the same brick are unlikely to be differently sourced; and yet they can be chemically different. Thus the heterogeneity or uniformity of the brick, and how it varies, must join the line of potential characteristics from which to learn about bricks. There is unlikely to be a silver bullet to find brick source from single analyses, no matter how many elements are measured in the analysis, because there is no single base for comparison. The distribution of answers is as important as any individual one in a heterogeneous system.

However, the heterogeneities are mineralogically recognizable. The presence or absence of accessory phosphates like apatite and ultra-refractory minerals like zircon is clearly a useful characteristic to track in the source materials of differing geographic provinces. In addition the detailed chemical characteristics of the accessory phases may carry interesting provenance information. For instance the phosphates in Dutch DUCY and Danish DAFY are quite different in their LREE enrichments. Both have similar Lu, but La in DUCY is depleted whereas La is quite enriched in DAFY phosphate. (See La-rich/La-poor markings in Figure 6.) Zircons also have the potential to show wide and interesting variations in their REE chemistry (Belousova et al., 2002; Grimes et al., 2015) which may be useful for determining provenance, although not



always (Hoskin and Ireland, 2000). It is unknown at present whether the U-Pb systems used for high precision age dating in zircons would be reset by the firing process in brick manufacture or not. There may be provenance information to be had there, which could become a promising avenue for further study, as it has been for sandstone sand particle provenance (Dickinson and Gehrels, 2003).

The parallel complementary tracks of particle information and bulk chemical information have been followed in the examination of ceramic pottery from Pacific island prehistoric cultures. Dickinson (2006) used the petrography of temper sand particles to pinpoint the island of origin of pottery, to establishing trade patterns in Oceania prehistory. The individual island sources of temper sand have distinct enough tectonics, volcanism, and hence particle types that individual terrains are possible to distinguish. It is worth noting that the island geology of the Pacific is not so intimately tied to the continental crust as that of the Atlantic. And indeed the chemical studies of Kennett et al. (2002, 2004) on prehistoric pottery from Oceania suggest that bulk chemistry may get a bit more traction in the provenance problem that it did in our study of continentally-dominated Atlantic bricks. A more global comparison of bricks from the Atlantic and Pacific basins should be revealing about such questions as: Does the signature of upper continental crust reach beyond areas where it is the prevailing bedrock? Do bricks made from the materials of volcanic arc and oceanic island terrains have a different background signal in their clay matrix than that of neighboring continents? These are questions for further study.

A final comment is that future investigators may wish to augment the base of knowledge of the details of the particle types comprising bricks of various provenances, ages, and manufacturing techniques. Such practices as the use of clinker and coal waste as in the 19<sup>th</sup> century Hudson Valley and Britain, or in contemporary China (Zhou et al., 2014), the use of distinctive sand and tempering aggregate with characteristic accessories like the placer mineral suite of heavy durable minerals, or the efficacy of the blending and bonding employed may provide further characteristics that could be useful for fingerprinting brick sources. Observations of this sort are not intrinsically difficult -- they are certainly easier than measuring age in zircon -- they simply require enough time and sustained attention to amass a sufficient data base for useful, comprehensive reference. Geologists have been successful in recognizing the sources of sedimentary rock deposits using these techniques, which very likely will be transferable to the study of brick sourcing, given a library of information to consult. Archiving of ceramic chemistry has been initiated by Gilbert et al. (1993) as a start on that sort of library. Progress may follow the addition of particle characteristics to such an archive.

### **Afterward**

Regrettably the question which initially motivated this study does not have a definite answer at present. We do not yet know the source of the bricks used in the construction of Ft.

Sint Kruis. In terms of brick dimensions and geographic commonality, the Curacao DUCY specimen was an early favorite for the Dutch-derived hypothesis. However the stark differences in the REE patterns for DUCY (strong HREE enrichment) vs. SC (HREE depletion) is a powerful incentive to look elsewhere. We have discovered that these stark differences are the result of a small but significant population of zircons in DUCY (and apatites in DAFY). If these accessory phases are analytically filtered by screening the TRA in the laser track to remove them, then DUCY, DAFY and SC are very similar. In fact this zircon population is not found at the opposite end of DUCY so that DUCY\* closely resembles SC, SC\*, and filtered DUCY, and filtered Danish DAFY! Without the accessory particles, bricks are poorly distinguishable in their bulk chemistry. The answer to “Who built Ft. St. Kruis?” is not at hand, but the direction in which to seek it is clearer: look at the particle population for fingerprints.

### **Acknowledgements**

This study owes its conception to the curiosity of William A Taylor, architect, of St. Croix in the Virgin Islands. We thank him and Dr. Charles Gehring of the New Netherlands Institute of the New York State library for providing brick samples, background information and commentary, and encouragement to pursue this study. We thank Roberta Rudnick, Terry Plank, and Alan Gilbert for their helpful comments on drafts of this report. This is LDEO contribution XXXX.

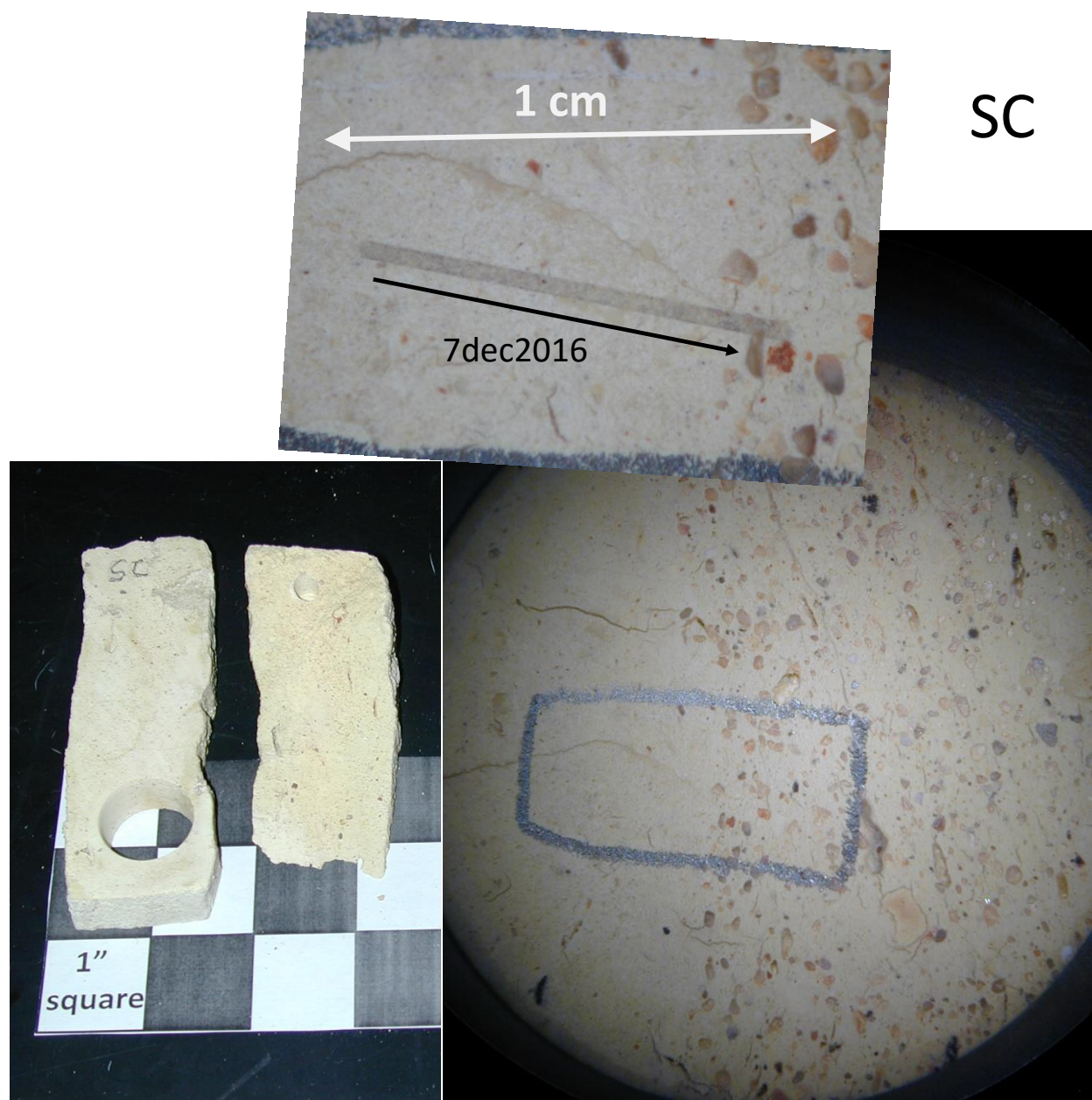
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### Appendix of documentation

One inch round mountings for 1<sup>st</sup> round [7dec2016] of LAICPMS analysis of 19mm core plugs and inset of laser track region. The analyzed surfaces are very heterogeneous. White double-ended arrow is cm scale bar. Black arrow indicates laser traverse direction. Macro-view insets shows the position from which the 19mm cores and the 6mm cores were cut. Additional background documentation interspersed.



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7 Sept 16

Dear Dave &amp; Nick,

These brick samples are possibly Dutch  
1620-1630's "Belgium Roman" 220 x 105 x  
40 mm, shipped as ballast from *Teiden(?)*  
in preparation of an naval attack on  
San Juan that lasted 6 weeks. The Dutch  
wanted to displace the Spanish from their  
capitol and used their closest base, Sint  
Kruis, to supply the fleet.

Dr. Charles Gehring, the director of the  
New Netherland Institute in Albany, will  
be sending another sample from Curacao  
that might match these. He is the ultimate  
authority on the Dutch in the New World.

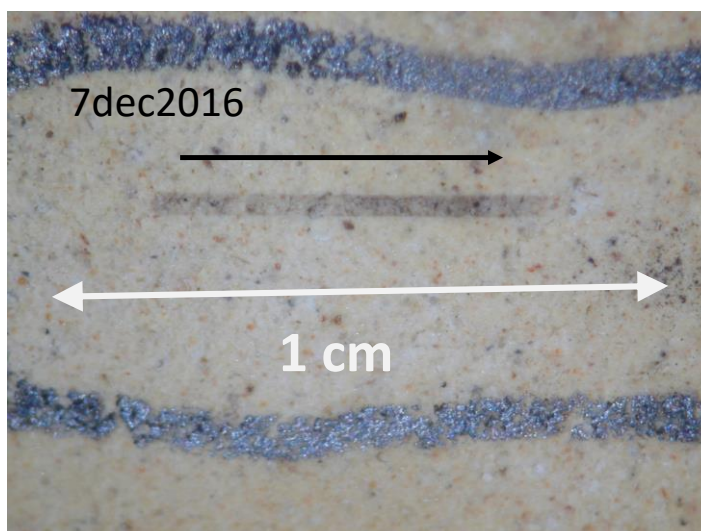
If the composition of these clay bricks  
match Dutch sources, that will confirm dating  
to 1625-?

Sincerely Thanks,  
Bill



# Background on brick SC





DUCY



### Report: Yellow Brick XRF Testing

One yellow brick collected from the Landhuis Santa Barbara in Curacao, and eleven yellow bricks and one red brick collected from three, early Dutch, archaeology sites in Albany and Manhattan, were tested using XRF. The purpose was to determine the elemental composition of the bricks and their approximate relative amounts, and then to compare the results to ascertain whether the source of fabric used to make the yellow bricks may have been the same.

The bricks were examined using a Bruker Tracer III handheld XRF analyzer without a filter for 60 seconds each. The elemental composition of the bricks was read on the machine's display unit, and the results were then transferred to a computer to be made available for digital documentation. Given the lack of homogeneity of the bricks, the XRF analyzer cannot analyze the quantity of elements present, but rather indicates through graphic representation their relative amounts.

The readings revealed that the predominant elements in each brick were iron (Fe) and calcium (Ca). The third noticeable element was silicon (Si), appearing consistently as a much smaller amount relative to the iron and calcium. The only other element that appeared in significant amount was chlorine (Cl), which was detected in only one brick.

It was expected that compared to the yellow brick, the iron content of the red brick would be higher, and it was. The ratio of iron to calcium in the red brick was nearly 7 to 1. In the case of the yellow bricks, it was expected that the ratio would be lower since calcium is an element that contributes to the 'yellowness' of this variety of brick. This was borne out: the average ratio of iron to calcium in 9 of the 11 yellow bricks tested was  $2\frac{1}{2}$  to 1. In the case of the other two yellow bricks, each of which had a heavy deposit of mortar on them, the average ratio of iron to calcium was  $\frac{1}{2}$  to 1. This, too, would be expected given the high calcium content of mortar.

The yellow brick from Curacao, for which this analysis was conducted, proved to be similar to, but not the same as, the 9 yellow bricks whose ratio of iron to calcium was  $2\frac{1}{2}$  to 1. It was tested in three places: two visually yellow locations, and the other, a reddish inclusion in the brick. The average ratio of iron to calcium in the first two locations was 1-1/3 to 1, and in the third was  $2\frac{1}{2}$  to 1. The unexpected element that appeared in all of the readings for this brick was chlorine. The average ratio of chlorine to calcium was approximately that of the iron: 2 to 1.

It would be difficult to conclude that the source of clay from which the yellow bricks were manufactured was the same. In the case of the brick from Curacao, the brick is considerably more deteriorated than the others, and from appearance, more heterogeneous than the others as well. Also - it's possible that having been located in a briny, tropical climate for several hundred years is a factor in the brick's deterioration as well as the obviously high reading of chlorine on its surface. This compares to the other bricks which were all excavated from an apparently more shielding environment in the soil of a temperate climate.

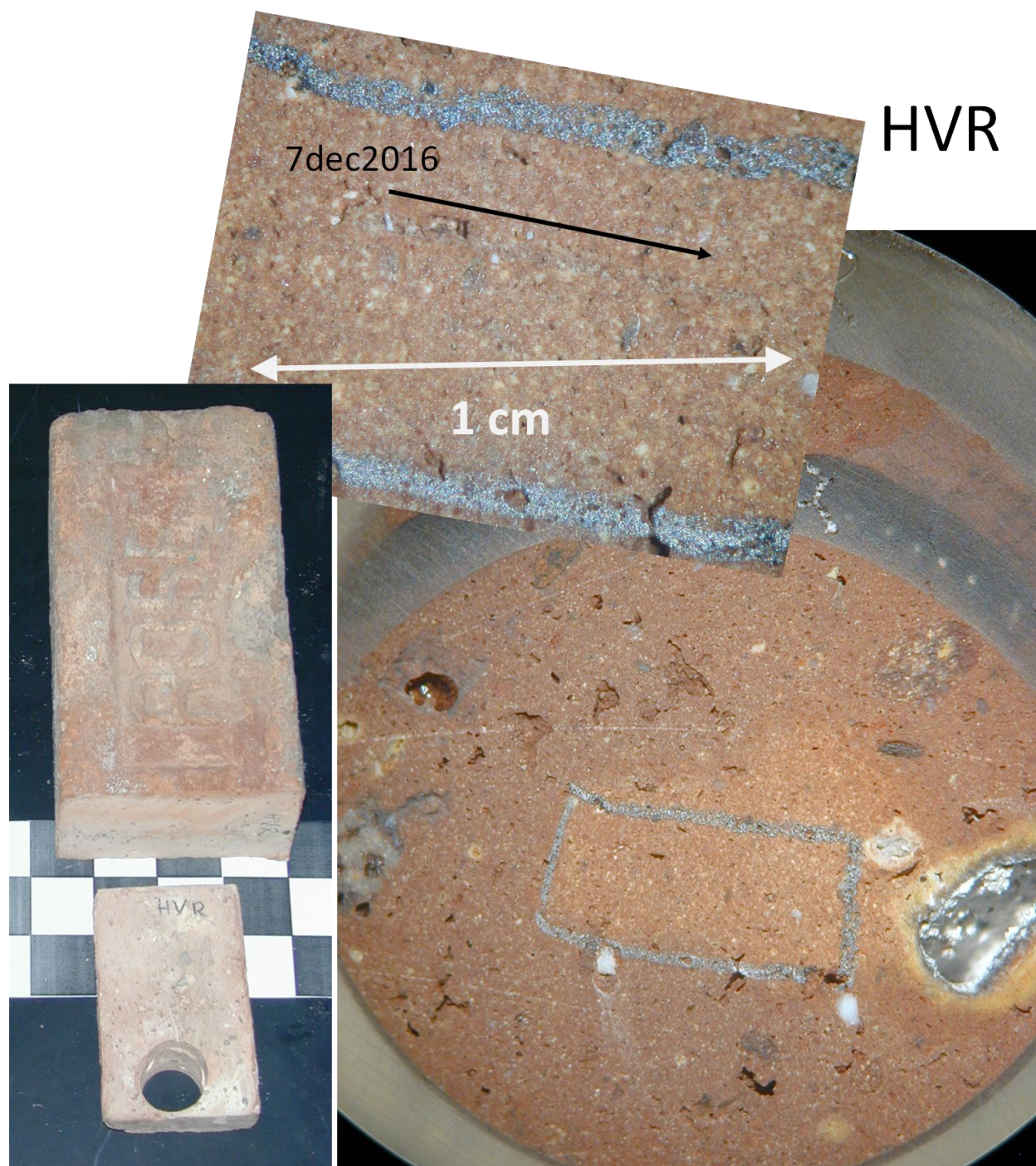
What can be said is that the yellow bricks we examined do have a higher percentage of calcium relative to iron in their fabric as compared to the red brick. To ascertain the source of the materials that were mixed to produce the yellow bricks, however, would require instruments capable of measuring and quantifying the elements, and a more sophisticated method and analysis that takes into account variables that can interfere with results.

### Background and XRF report on brick DUCY from Dr. Charles Gehring

"Ysselsteen" 2/28/87  
From  
Landhuis Santa  
Barbara on Curacao.  
Brick brought over  
from the Netherlands  
to be used in archways  
etc. or wherever strong  
building materials  
were required.  
Gov. Rönner said that  
brick came from oldest  
section of buildings.

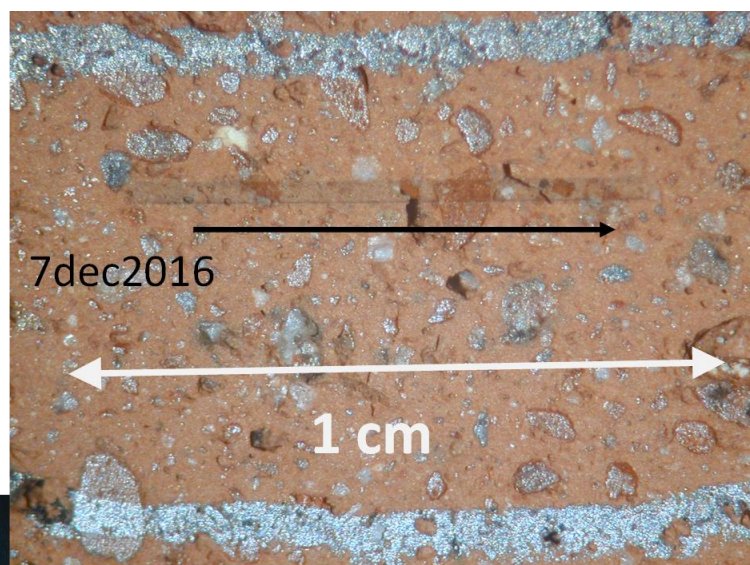






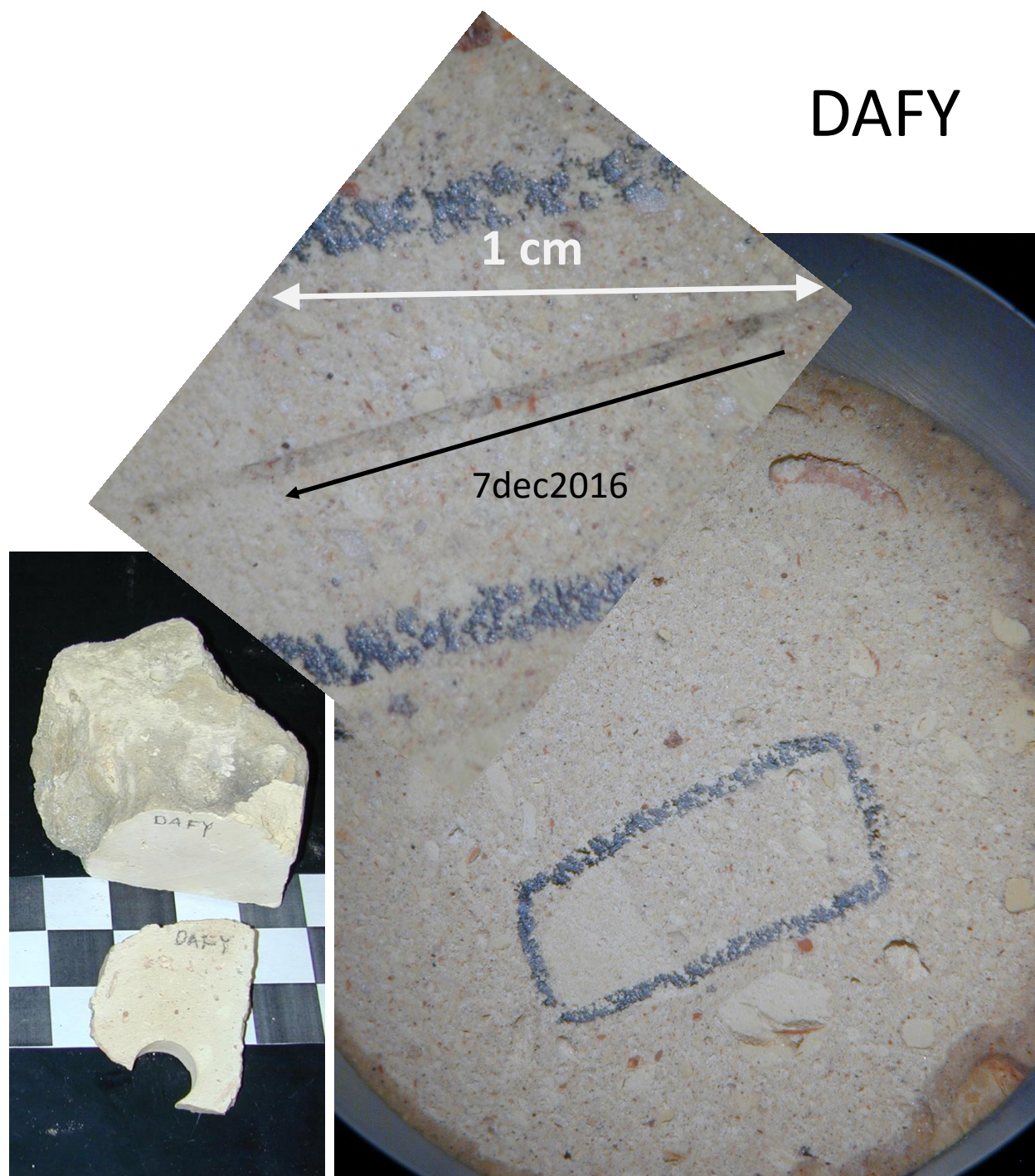


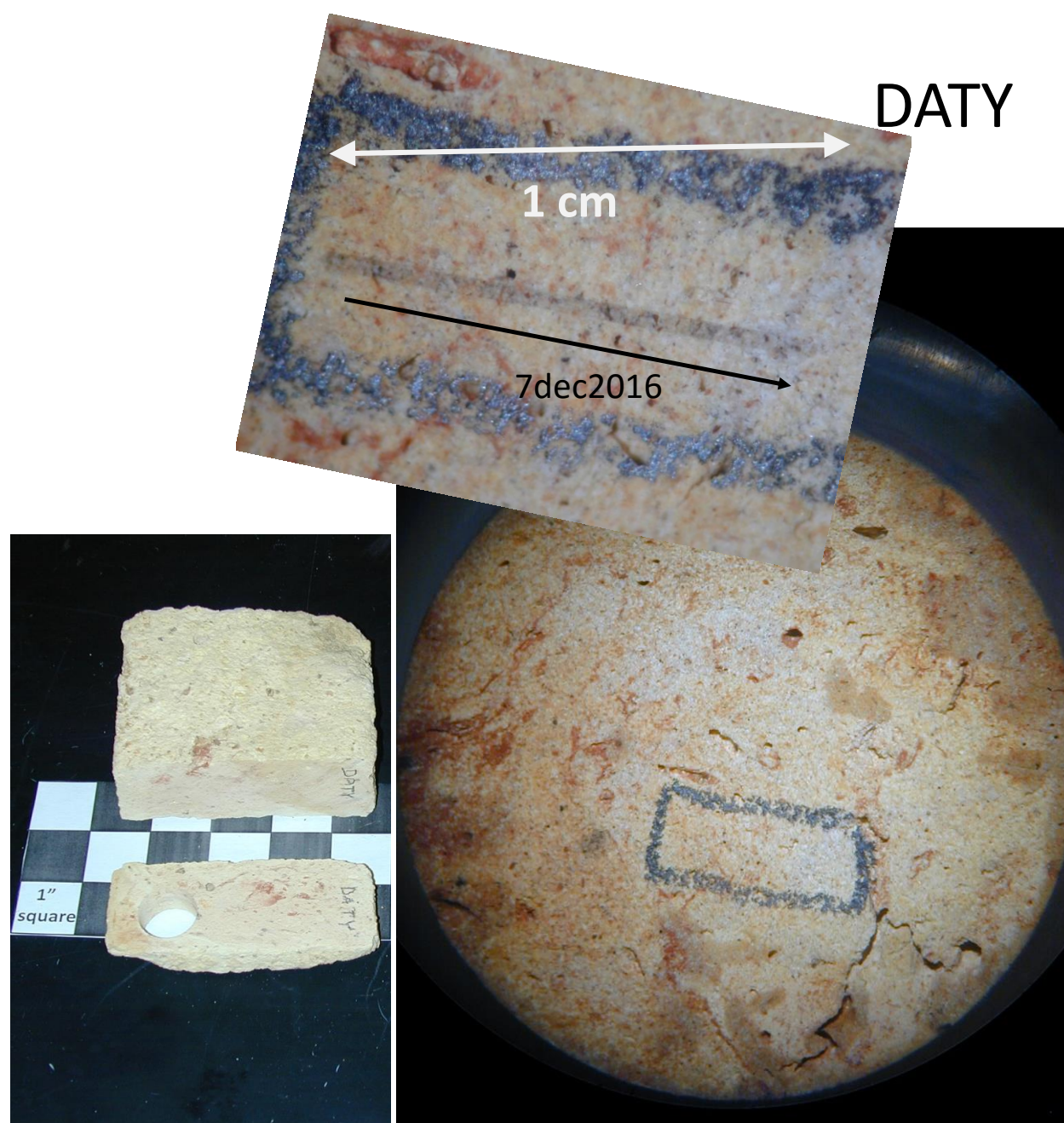
# HVCX



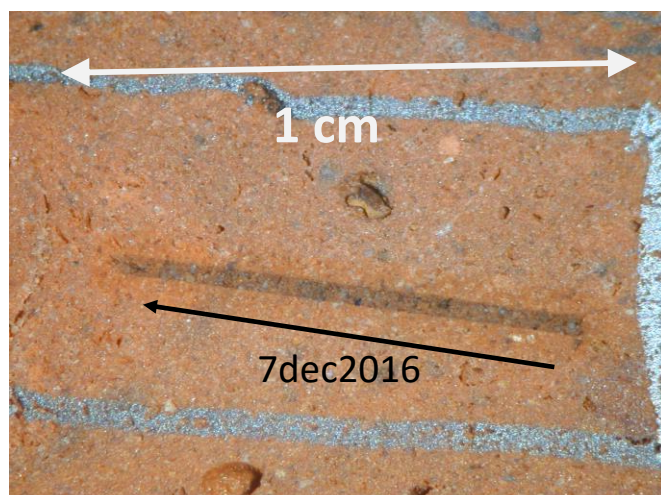


# DAFY



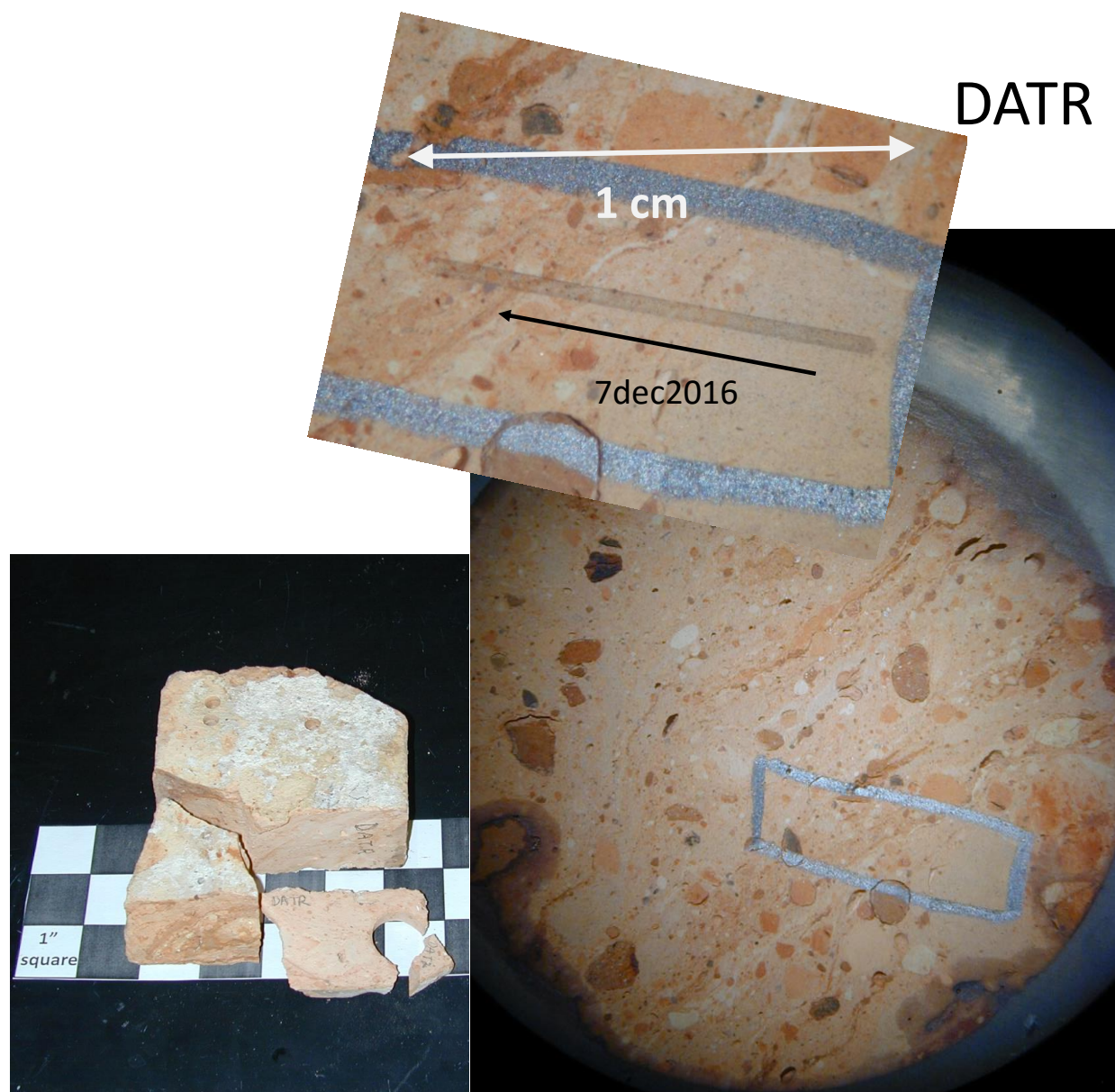






DAFR







## Background on Danish Bricks

15 Oct 16  
DAVE  
2 classes of "fat" Bricks, red & yellow  
appear to be "OLDENBURGER FORMAT (OF)  
the thinner, yellow & rose color  
appear to be "Belgium Roman" by  
the Dutch classification System  
(S.I.) Systeme International  
d'Unites. This doesn't mean they  
are Dutch rather than Danish. These  
samples are all Danish 18<sup>th</sup> C.  
Dr. Gehring's sample is certainly  
Dutch 1630's. Sint Kruis could also  
be 1630's but the Dutch were here, on  
and off in the 1620's could have  
built Ft. Sint Kruis the same time as  
Curacao. All bricks are 8.75" L x 7".  
Rgds, Bill

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Sint Kruis



