Medieval Gunpowder Research Group

Making saltpetre

Report Number 11 – September 2013

Middelaldercentret, Nykøbing, Denmark

Introduction

One of the main aims of the Medieval Gunpowder Research Group is to make the ingredients of gunpowder by methods based on medieval or early modern sources. This would improve our ability to investigate and better understand the properties of medieval, as opposed to modern, industrially produced, gunpowder.

Of the three ingredients, the easiest to produce was the charcoal which we have made in the traditional way – technique which has not changed for centuries (see Medieval Gunpowder Research Group, Report 1, September 2002). Slightly more difficult was the production of sulphur but we have collected sulphur from the sites where it was mined in the medieval period and refined it sufficiently to produce black powder (see Medieval Gunpowder Research Group, Report 1, September 2002 and Report 5, 2006). However, it is the saltpetre which has proved to be a problem and our earlier attempt was a failure (Medieval Gunpowder Research Group, Report 3, June 2004). This failure is due to the complexity of the process and the associated problems of the interpretation of the original sources and texts. While much of the method is clear enough there are two areas which are open to interpretation. The first is what a saltpetre bed was made of and the second is the refining process of the intial product from the saltpetre bed.

Our work in 2013 focussed on investigating these two problems and to finding solutions to them. We would then be able to propose a method to produce a significant quantity of good quality saltpetre for use in further experiments.

The saltpetre bed – a brief historical survey

The earliest sources to deal with the subject of gunpowder are the Feuerwerkbucher produced throughout the 15th and early 16th centuries. Though they contain much about gunpowder mixtures, incendiaries and the firing of cannon they do not contain a great deal of information about the making of the three ingredients used for gunpowder. There are some descriptions of how to make saltpetre but these do not seem to be methods that would actually work (Smith 2010). For example, this method of using the walls of cellars:

> *If you want to grow good saltpetre on walls, pour saltpetre water in which saltpetre has been boiled against the moist walls of a cellar or wherever saltpetre prefers to grow. This wall will produce enough saltpetre and when you have removed it, always sprinkle water against the wall so that it stays wet. It [saltpetre] likes to grow thus.*

This method has been tried and found not to work, producing sulphate and not nitrate (Smith 2010). The likely reason for it not working is that there is no source of nitrogen.

> *A second description for making saltpetre was to 'grow' it on a clay tube;*
> *Have a tube made [of porous clay], as large as you wish, which is full of small holes. Take a pound of tartar and half a pound of common salt (or as much as the tartar), but three times as much limestone and the urine of a man who drank wine. Make a thick paste from this and spread it on the tube inside and outside and then let it stand in the sun for three days. On the fourth day remove the paste from it and hang the tube in a cellar. From this good saltpetre will grow.*

Again we have to ask whether this method would actually work. Tartar is probably the salt potassium bitartrate or potassium hydrogen tartrate, which has the formula KC4H5O6. This contains the necessary potassium for the saltpetre. Unfortunately the addition of common salt, sodium chloride, would not be a good idea as this would become a detrimental impurity in any saltpetre produced. Limestone, that is calcium carbonate, would help to keep the mixture alkaline and might be beneficial. Finally the addition of urine would certainly add a necessary

raw material for saltpetre – a nitrogen compound, urea. Strangely the method goes on the say that you have to then remove the paste before it will produce saltpetre. It is difficult to understand just how this method would produce any nitrate and it is easy to dismiss it as nonsense. However that this method was used is reinforced by other similar descriptions and from illustrations. It is possible that the description is not complete and that something vital is missing though it is difficult to see what.

However, by the 16th century we are on much firmer ground and the written descriptions for making saltpetre make more sense. In 1540, Vannoccio Biringuccio, a 'master craftsman in the practices of smelting and of metalworking', wrote on saltpetre in his famous book, *Pirotechnia*. He says:

> *[saltpetre is] extracted with fire and water from arid and manurial soils, from the growth which exudes from new walls or from that loosened soil that is found in tombs or uninhabited caves where the rain cannot enter. It is my belief that it is engendered in these soils from an airy moisture that is drunk in and absorbed by the earthy dryness.*

He then goes on:

> *The best and finest of all saltpetres is that made of animal manure transformed into earth in the stables, or in human latrines unused for a long time. Above all the largest quantity and the best saltpetre is extracted from pig dung. This manurial soil, whatever kind it may be, should be well transformed into a real earth and completely dried of all moisture; indeed, it should be powdery if you wish it to be good.*

In 1580 Lazurus Ercker published his Treatise on Ores and Assaying which includes a long section on saltpetre. He writes:

> *The best earth, which is very rich in saltpetre and does not contain much salt, is the earth from old sheep pens that have rotted from disuse, provided that it is very dry and not wet. Another kind of earth, which also yields good saltpetre, is the lime or mortar from very old walls that have stood in a hamlet or in a city where the soil of the land itself is saltpetrey and where they have been neither too wet with rain nor continuously dry; instead, they should have been moistened at intervals and then exposed again to sunshine. The next best earth comes from broken plaster walls, where buildings have been razed and the resulting rubble has been dumped into vaulted cellars and has been lying there for several years. The earth is supposed to be good for the reason that the mustiness from the cellar seeps into it through the vault and it is additionally moistened from above by the daily sprinkling, and saltpetre is thus generated therein. This kind of earth can be dug up to a very great depth, since it is good from the top down to the vault. . .*
>
> *All earth from buildings with dirt floors, cellars, or rooms that are old and have been unused for a long time is also good. But where it does not lie over a cellar, it is not advisable to dig it up more than a span or half an ell deep without assaying it. However, it should be remembered that the regions of the country where you wish to boil saltpetre must themselves be saltpetrey or must have a natural tendency for the generation of saltpetre. Mostly these are regions in good, level, fat, and loamy country.*
>
> *All earths from horse stables with dirt floors are suitable for the boiling of saltpetre, as well as old rotted garbage dumps outside the cities after they have been spaded up so that the sun can have its effect on them, old latrines, the earth from breweries and dye-houses and from those places where alum-bearing, unctuous substances are handled, together with the used ashes that are dumped outside cities by soap boilers and tanners, and all other ashes from which lye is made. However, almost all such earths yield much salt, which constitutes a disadvantage in boiling.*

Saltpetre works – from Ercker.
A The front part of the saltpetre works, which houses the leaching vats
B The back part, which houses the boiler, and where the boiling takes place
C The old beds from which saltpetery earth is scraped
D Wood for the boiler
E The workman who scrapes the earth from the old beds

Again, like the description by Biringuccio, it is obvious that Ercker clearly understood just how to make saltpetre. Perhaps the best early description is given by Gerard Honrick in 1561. Honrick offered offered to sell the secret of how to make saltpetre to the English for £300 – an enormous sum of money. His method was very similar to those of Biringuccio and Ercker and provides a great deal of detail – see Appendix 1 for his full account. In essence his method used four basis ingredients, earth, urine, dung and lime. Now we need to consider how a process like this might work.

Our saltpetre bed

It is clear then, that making saltpetre is a complex process and one which is not completely understood. However, from this brief survey of the literature, our modern chemical knowledge and the few experiments which have been conducted on the subject we are now more certain of the process of how to make saltpetre, potassium nitrate, KNO_3. First a source of nitrogen is needed and second, a way to convert that nitrogen into a nitrate (NO_3).

Although nitrogen, N_2, makes up almost 80% of the air we breathe and is a vital element in all living tissues, it is a very inert element and does not easily combine with other elements to form compounds. This means that nitrogen, apart from its presence in the air, is not often found in nature – you cannot go and dig up a pile of nitrates or sink a mine to find it. Strangely, though, nitrogen compounds can be produced by living organisms. For example peas and beans can 'fix' nitrogen from the air and form compounds from it, which is why they are grown as a way to enrich the soil. Animals, including humans, also produce nitrogen compounds as part of the process of breaking down the food they eat. Initially, ammonia, is formed but due to its toxicity, our bodies immediately convert it into another, non-toxic, nitrogen compound called urea, which is then expelled from the body in urine. Urea is made up from nitrogen with carbon, hydrogen and oxygen, its chemical formula is $(NH_2)2CO$. It is this compound, urea, which is fundamental to the making of saltpetre.

So the urea in urine is the source of the nitrogen in saltpetre but it still has to be converted into nitrate, NO_3. This is where the soil and dung come in. They contain bacteria which can break down the urea and convert it, eventually, to nitrate. The first step in the process, the conversion of urea to ammonia, is carried out by bacteria in soil which contain the enzyme urease. The ammonia is then oxidised by other bacteria, nitrosomonas to nitrite and another bacteria, nitrobacter, converts the nitrite to nitrate. The process can be summarised:

1. Urea $((NH_2)_2CO)$ + bacteria (urease) → ammonia (NH_3)
2. NH_3 + nitrosomonas → $NO^{2-} + H_2O + H+$
3. NO^{2-} + nitrobacter → NO^{3-}

In order to investigate this process, Williams (1975) carried out a series of experiments which provide good supporting evidence. First he made two piles of fresh cow dung each of approximately 18kg. To one he added 3kg of slaked lime and left the piles for a year. No nitrates were found in either pile. He then added 2 litres of urine to the piles each week and found that after 3 months a small amount of nitrate was produced. He also tried leaving about 20 litres of urine for several months and found no nitrates were produced. Finally he prepared a further two piles consisting of 20kg cow dung with 20kg of soil, to one of which he added 3kg of slaked lime. He then added urine and took samples from each pile for 18 months. After a period of some 6 months both piles started to produce nitrate and after 18 months the final analysis gave figures of 14,900 ppm for the pile with lime and 16,550ppm for that without. His conclusions are worth quoting in full:

1 Traces of nitrate form first in the tops of the piles, then percolate downwards so that the concentration is similar throughout each pile. This would explain why Honrick's recipe contains elaborate instructions for spreading the materials into thin layers. The same effect would be obtained by turning over the piles frequently

2 Both urine and dung are essential (something the early printed recipes purposely do not make clear) and the admixture of earth is also necessary to obtain an appreciable yield. Traces of calcium carbonate are also desirable, but quicklime specified probably reduces the initial yield.

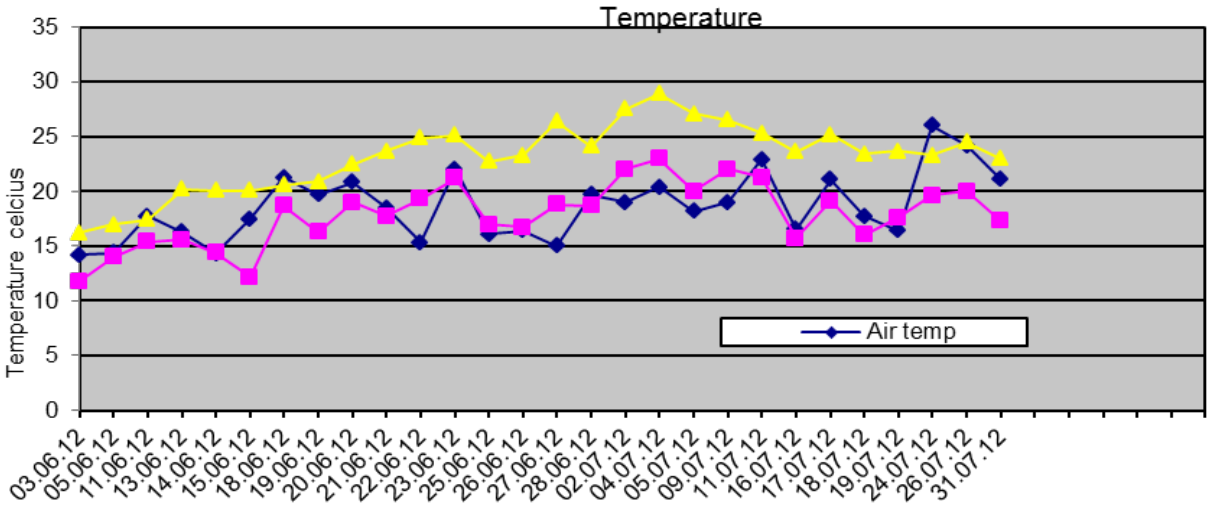
3 After 1½ years, the maximum yield is still only of the order of one per cent. Even allowing for an increase in reaction velocity if thin layers were employed, it can never have been a rapid or efficient process. It is remarkable that it was ever devised at all, and the author, for one, has acquired a considerable respect for the powers of observation and experimental skill of the 14th century chemists who discovered how to operate the artificial nitre-bed. (Williams 1975: 133)

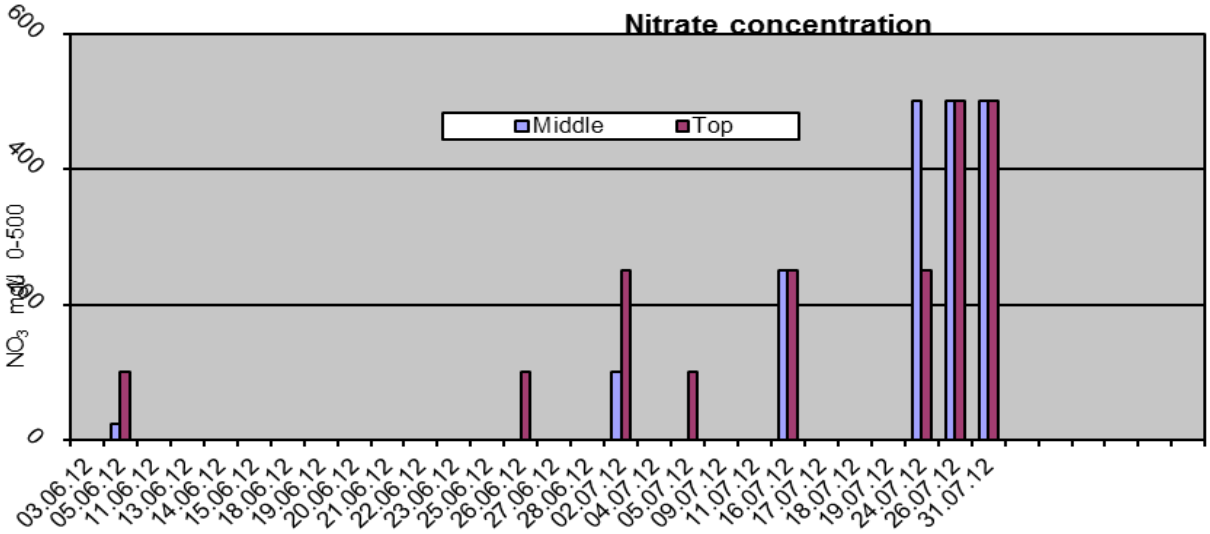
This evidence led us to propose a new saltpetre bed made from animal dung, soil and urine and in early 2012, a bed like this was made at the Medieval Centre. A simple shelter was built consisting of a wooden back wall with a lean-to style roof open at the sides and the front. A pile of roughly 500kg of soil and 500kg of pig dung was made under the shelter and urine was added on a monthly basis when the pile was also turned to ensure it was aerated – oxygen is a key component for the bacteriological processes. Once the pile was completed, 50 litres of urine were added every month from May to October 2012 and 50 litres added every 14 days from May to September 2013.

The saltpetre bed at the Medieval Centre.

A number of measurements of the pile were taken between the beginning of June and the end of July 2012 in an attempt to check that the process of making nitrate was under way. The temperature of the top and middle of the pile as well as the air temperature, the nitrate and pH level at the top and middle of the pile. The nitrate level was measured using nitrate indicating papers. The results are tabulated below:

	Temperature ^{O}C			*Nitrate concentration mg/l*		*pH*	
Date	*Air*	*Top of pile*	*Middle of pile*	*Middle of pile*	*Top of pile*	*Middle of pile*	*Top of pile*
03.06.12	14.20	11.80	16.20				
05.06.12	14.40	14.00	17.00	25.00	100.00	10.00	10.00
11.06.12	17.70	15.40	17.40				
13.06.12	16.30	15.60	20.20				
14.06.12	14.30	14.40	20.10				
15.06.12	17.50	12.20	20.10				
18.06.12	21.30	18.70	20.60				
19.06.12	19.70	16.30	20.90				
20.06.12	20.80	19.00	22.50				
21.06.12	18.50	17.70	23.70				
22.06.12	15.30	19.30	24.90				
23.06.12	22.00	21.20	25.10				
25.06.12	16.10	17.00	22.70				
26.06.12	16.40	16.70	23.30	0.00	100.00	11.00	10.00
27.06.12	15.00	18.80	26.40				
28.06.12	19.70	18.70	24.10				
02.07.12	19.00	22.00	27.50	100.00	250.00	7.00	5.00
04.07.12	20.40	23.00	28.90				
05.07.12	18.20	20.00	27.10		10.00		7.00
09.07.12	19.00	22.00	26.50				
11.07.12	22.90	21.30	25.30				
16.07.12	16.50	15.70	23.60	250.00	250.00	11.00	8.00
17.07.12	21.10	19.10	25.20				
18.07.12	17.70	16.00	23.40				
19.07.12	16.40	17.60	23.70				
24.07.12	26.00	19.60	23.30	500.00	250.00	9.00	9.00
26.07.12	24.20	20.00	24.50	500.00	500.00	7.00	7.00
31.07.12	21.10	17.30	23.00	500.00	500.00	9.00	9.00





The temperature in the pile was above that in air indicating that some form of reaction was taking place. This was confirmed by the nitrate concentrations. The pH of the pile was also greater than pH7, a condition for the reactions that produce nitrates. All this evidence supported our hope that the pile was producing nitrate.

A sample of the saltpetre earth showing white crystals on the surface

Preliminary extraction experiment

In early September 2013, 10 litres of water were extracted through a part of the pile. This was boiled down to about 150ml and left for 3 days. Long, thin brown coloured crystals precipitated out which were probably saltpetre as it is known to form long, needle like crystals.

Crystals formed from preliminary extraction

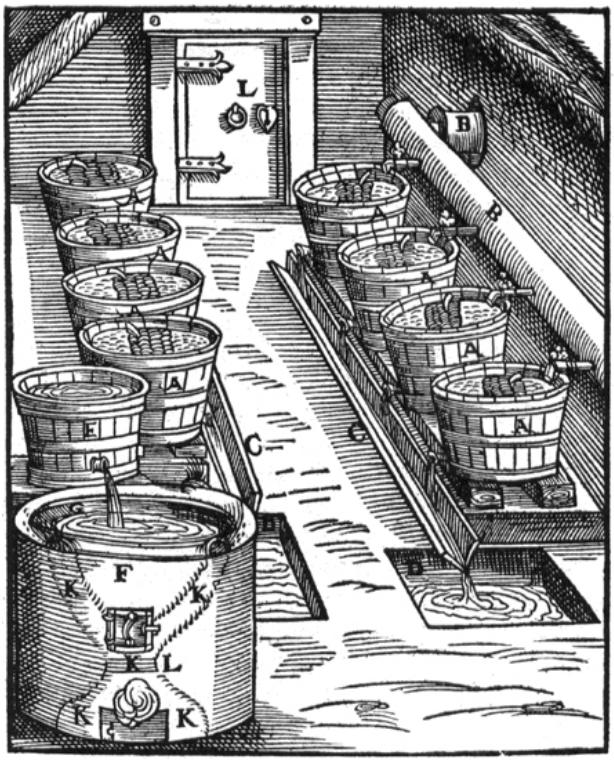
Detail of crystals

This preliminary extraction gave us cause to hope that the pile had produced saltpetre – something confirmed by mixing a small amount of these crystals with sulphur and charcoal – a mixture which burned fiercely when lit.

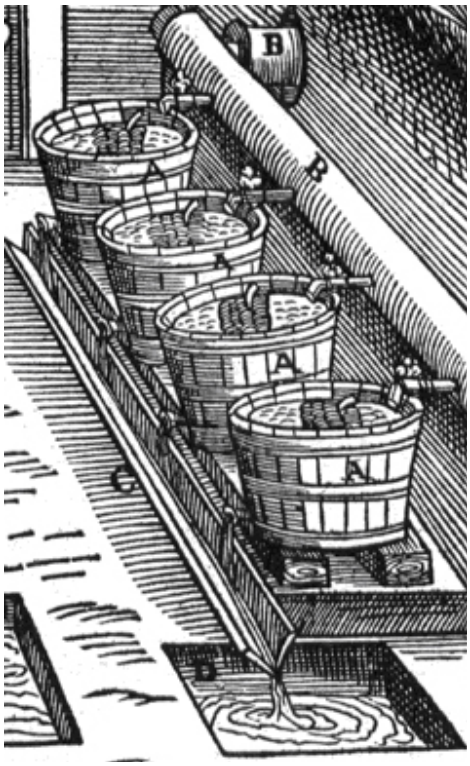
The main extraction

We now had what we hoped was a good source of saltpetre and the next problem was to extract it from the soil and dung. This process is reasonably well documented and understood as the historical process makes sense in modern terms. However we wanted to investigate the various issues thrown up by the descriptions and try them out ourselves.

The extraction of saltpetre from the bed was carried out over a week in September 2013. The method was largely based on the work of Ercker in 1580 and the subsequent 'reworking' of his method by Thomas Henshaw in 1667.



The method extracting and boiling down. From Ercker



Detail showing the tubs of earth being extracted. The wicker mats (rundles) can clearly be seen

We carried out the extraction using a large plastic tub able to hold about 40 kg of earth and we completed 10 extractions. First, a small hole was drilled in the bottom of the tub and this was plugged with a wooden plug. We then prepared each tub in the following way:

A layer of small twigs was laid in the bottom of a plastic bucket to prevent the soil blocking the drain hole.

This was followed by a generous layer of straw.

Then, between 1 and 2 kg of wood ashes were added.

About 40 kg of the saltpetre earth was carefully laid on top.

Finally 30 litres of water was added

The tub was the left for a period of time before the plug was removed and the water with the dissolved salts was allowed to drain into a bucket.

The gas boiler

Boiling the liquid down - the scum formed on top was continually scooped up and removed

The extracted liquid was then poured into a gas fired boiler to enable us to boil the liquid down. In order to check that there was nitrate in the liquid a simple check was made using a nitrate indicator paper – all readings were in excess of 80 mg per litre of water.

Although this basic procedure was carried out each time, we altered the timings and procedure as a means to investigate the process as well as in response to discussions and thinking as the process proceeded. The following summarises the 10 extractions we carried out.

1 40 kg of the pile with 30 litres of water. Left for an hour,

2 40 kg of the pile with 30 litres of water. Left for an hour. [this bucket broke and was sat inside another bucket]
We put both of these together and boiled down to about half in the gas boiler – we then took about 18 litres of the boiled down liquid and put in second, electric boiler,

3 40 kg of the pile with 30 litres of water. Left for a short period, drained and added to the gas boiler.

4 40 kg of the pile with 30 litres of water. Put in extra straw – a thicker layer. Tub 4 was left overnight – liquid was quite a bit clearer than before with less turbidity and fine silt in it.

5 40 kg of the pile with 30 litres of water. Putting in a thick layer of straw with all now. Two fresh buckets of water run through this tub and this used as the water for tub 6.

6 Repeat of 5 – using water from 5 with a little extra to make 30 litres

7 Repeat of 5 – using water from 6 with a little extra to make 30 litres

8 Repeat of 5 – using water from 7 with a little extra to make 30 litres

9 Soil from pile broken up by forcing through a simple sieve – then repeat of 5 using water from 8 with a little extra to make 30 litres. Left overnight

10 As 9 – using water tub 9

On the morning of day three, 5 litres of the liquid (tubs 1 to 8) was put in an electric boiler and reduced to approximately 500 ml. Small portions were let off into aluminium trays and left to cool/precipitate – called 1 to 4. Tray 4 was the last portions removed. We then continued to boil the liquid down, adding the leachate from tubs 9 and 10 when they were ready. We continued boiling down till we had about 500ml of a light brown liquid and we left this to precipitate out.

The final boiled down liquid being run into a tray for precipitation

Small needle like crystals started to precipitate out of the solution and these were collected. In total we obtained about 105g of saltpetre.

The needle-like crystals of saltpetre

Close-up of the crystals showing their needle-like form

This final product still appears to be quite impure and needs further refining. We are hoping that we can have a sample analysed.

Observations and suggestions for future work

The appearance of the liquid extract varied considerably from a relatively clear pale brown liquid to a dirty, darker brown. This appeared to be partly related to the amount of time the water was left in the tub – the longer the clearer it was. However it was also clear that a lot of fine earth was making its way down to the bottom of the tub and was coming through due to the hole being very close to the bottom of the tub. For future work we think it better if the hole was on the side of the tub, 2-3 cms up from the bottom so that less of the fine soil would escape. It would be good to have a side tap as in the illustrations. This problem could also be lessened by the use of a false bottom to the tub as noted in Henshaw:

> *On the inside of the tub, near the tap-hole, you must carefully place a large wad of straw, and upon that a short piece of board, which is to keep the earth from stopping up the tap-hole*

Ercker also notes this and says:

> *… put in each one [vat] a perforated wooden bottom, which, however, should rest as much as two fingers above the bottom [of the vat].*

Both Ercker and Henshaw recommend the use of a 'rundle of wicker, like the bottom of a basket, and about a foot in diameter'. This will 'keep the water, when it is poured on, from hollowing and disordering the earth.'

In digging up the earth we noted that it was quite lumpy and that there were large pieces of it sticking together. We realised that there would be a better extraction if the earth was broken down somewhat. We therefore used a simple plastic sieve to break it down into smaller pieces.

Another point which was clear was that we were not filling the tubs sufficiently with water and that there was a possibility that we were not extracting as much as was possible from the

Sieving the raw saltpetre earth to break it down into smaller pieces

earth. Careful reading of the texts again suggest that we proceed in the following manner, as Henshaw notes, 'Then pour on your earth common cold water, till it stand a hands breadth over the earth' he also recommends the use of a 'stirring stick' or cudgel, '…stick into the earth a good strong cudgel, which must be thrust pretty near the bottom…' This he notes '… is to be stirred about, to give the water ingress to the earth upon occasion.'

We had worried that this would create too much of a disturbance and would result in a great deal of the earth getting through to the tap hole. However Henshaw recommends leaving the tubs for 'eight or ten hours', more time than needed to dissolve the salts in the earth but enough for the earth to settle out of the water and leave it much cleaner. Creating a false bottom of wood, having the drain hole to one side and leaving the tub to settle for 8-10 hours would all mean that we would extract as much as possible from the earth and get a cleaner and less dirty material to boil down.

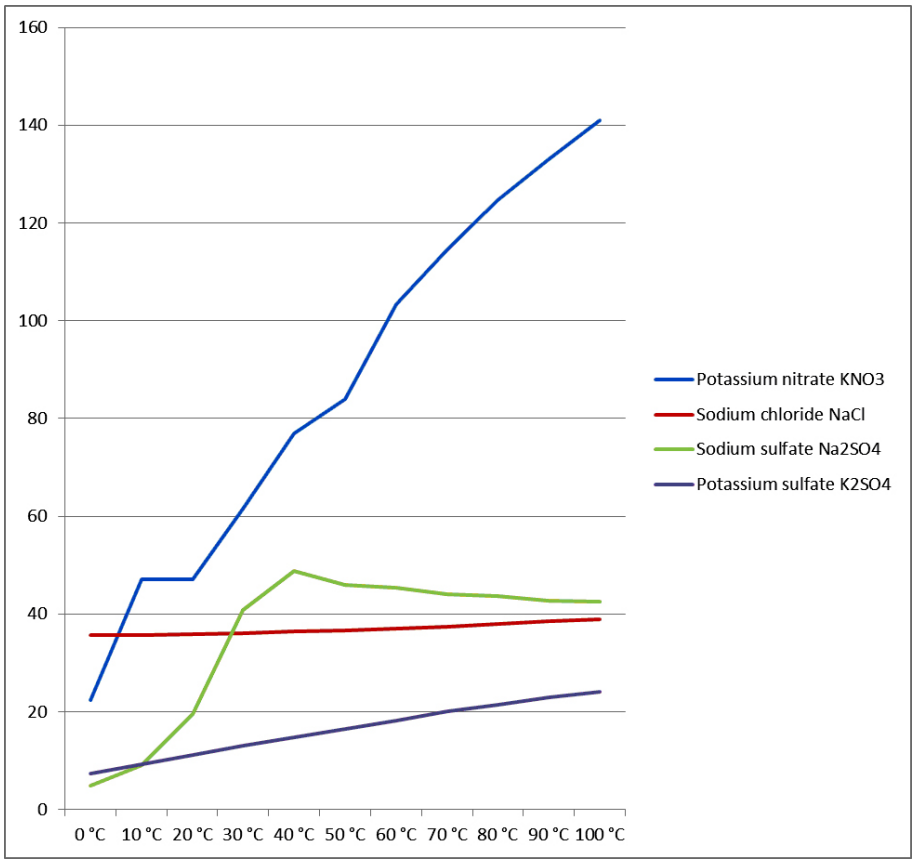
Both Ercker and Henshaw also recommend that water is put through each tub twice – the first will have a higher concentration of salts but there will be some that is not dissolved. Putting a second lot of water in and then using this as the first water in the next tub makes a lot of sense.

Purification

It took us some time and much discussion to fully understand the method of purification. Essentially the process depends on the difference in solubility at different temperatures between saltpetre and the other salts which will be extracted from the saltpetre bed and will be impurities. Most of these latter salts have roughly the same solubility in cold and hot water. For example common salt, sodium chloride, will dissolve 36g per 100ml of water at 20°C and 39g per 100ml at 100°C. However, the solubility of saltpetre is very dependent on temperature, ranging from 47g/100ml of water at 20°C to 141g/100ml at 100°C.

Table of solubilities of various salts in g/100ml water

Substance		0°C	10°C	20°C	30°C	40°C	50°C	60°C	70°C	80°C	90°C	100°C
Potassium nitrate	KNO_3	22.4	47	47	61.6	77	84	103.4	114.6	124.6	133	141
Sodium chloride	NaCl	35.6	35.7	35.9	36.1	36.4	36.7	37	37.5	37.9	38.5	39
Sodium sulfate	Na_2SO_4	4.9	9.1	19.5	40.8	48.8	(46)	45.3	(44)	43.7	42.7	42.5
Potassium sulfate	K_2SO_4	7.4	9.3	11.1	13	14.8	(16)	18.2	(20)	21.4	22.9	24.1



The solubities of the salts shown in graph form

This means that, if the raw saltpetre from the extraction, with all its impurities, is added to boiling water the latter will fall out of solution as their maximum solubility is exceeded leaving a solution of mainly saltpetre. The process depends on having a quantity of water at boiling point and then adding the raw saltpetre to it till the solution is saturated. On cooling, most of the impurities will then remain in solution while the saltpetre, with its much lower solubility at lower temperatures, will precipitate out. The saltpetre will, of course, contain some impurities but these will be greatly reduced and a second purification will reduce them a little more though it is impossible to remove all impurities by this method.

Pans and tubs for crystallisation – from Ercker.

A – The tall, slender vat in which the concentrated liquor cools
B – The furnace containing the boiler
C – The master who prepares the concentrated liquor and, removing the salt with a ladle, puts it in a little basket resting over the boiler on rails so that the strong liquor that was left on the salt can drain back into the boiler.
D – The little basket on rails
E – The small vat from which strong liquor runs into the boiler
F – The pans in which the crude saltpetre crystallises
G – The four tubs sunk in the ground, in which the crude saltpetre crystallises
H – A strong vat into which the liquor is poured that remain after crystallisation

We think that the process should be carried out in the following manner. First a quantity of water is heated to near boiling point. The raw saltpetre from the extraction is then added until the solution is saturated with saltpetre – that is, there is about 141g of saltpetre in every 100ml of water. Just when this point is reached in not easy to determine. Indian saltpetremen used a hydrometer and said it was ready when the specific gravity is 1320 though they also judged it by experience.

As more and more of the raw saltpetre is added, the amount of the impurities increases, but, as their solubility is still quite low at high temperatures, their concentration goes over their limit of solubility and any extra precipitates out of the solution and falls to the bottom of the vessel. There then comes a point when the solution is saturated with saltpetre, holding 141g/100ml, and with the salts of the impurities, but at much lower concentration. When this solution is allowed to cool, the saltpetre precipitates out, as its solubility decreases with lower temperature, with a small amount of impurities - much less than the amount in the saltpetre before the purification process.

Unfortunately we did not make enough saltpetre to be able to carry out this process but we think that this method is that descried by the sources as well as being the way that it was carried out in India (see Medieval Gunpowder Research Group, Report 6, September 2006)

Future work

The experimental work carried out in 2013 was extremely useful in that it helped us to work out just how the process of saltpetre extraction should be carried out and how we should proceed in the future – hopefully in 2014 when we can extract enough pure saltpetre to experiment with.

A summary of the process from Henshaw together with a suggested timetable is given in Appendix 2

The members of the HO Group, 2013

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Haileigh joined the HO group on a Society for the History of Alchemy and Chemistry (SHAC) New Scholars Award

Appendix 1[1]
Gerard Honrick's recipe for 'making of saltpeter' of 1561[2]

THE TREW and perfect arte of the making of SALTPETER to growe in Cellars, Barnes, or in Lyme or Stone quarries. The nature of SALTPETER is to growe in placis could, and drye, where neither sonne nor rayne entrithe, nor springe resort, for the dryer and coulder the place be, the [soner] and better do they bring forthe saltpeter.
And to MAKE the MOTHER OF SALTPETER these things following be requisite. FYRST black earthe the blacker the better. The next is URINE, namely of those persons whiche drink either wyne or strong bears. Then DONG specially of those horses, which be fed with ootes, and be always kept in the stables. The fourth is LYME made of plaster of Parys. The LYME whiche is made of OYSTER SHELLIS is the best, and better than th'other if it be kept from Rayne and water. The LYME whiche is made withe other STONES is nothing worthe for this purpose because it bindethe to moche, and bathe no saltness in it.
And to th'intent that those thinges may be conveniently orderid, firste th'earth is to be made as drye as duste and the stones are to be sifted oute. Then the Urine is to be stilled, throughe FRESH HORSDONG and the LYE coming therof must be kept in greate FATTIS or HOGS-HEADDES wherin muste be putte, a fourthe part of LYME UNSLEKT and one pounde of course or REFUS SALTPETER and th'earthe must be watered withe the [same] LYE in like sort, as we water lyme or morter, whiche we use for building, all which thinges must be well mingled together.
The Saltpeter is mingled withall, to th'intent it may the soner [receive] forme to growe: and after that the same is once put to youe shall not nede afterward to adde any more, notwithstanding that the said EARTHE may be occupied as long as your wyce.
And to the forsaid HORSDONG which is before steepid, let there be a greater quantite of FRESHE HORSEDONG added: and after it be so blended, let the same be mixt withe the EARTHE for when it is putrified it doth give drinesse, and heate to the EARTHE: and because SALTPETER proceadeth of drines, and couldnes, and those kinds be by nature could, and drye; that is to saye, TH'EARTHE AND URINE could: THE DONG: hoate and drye; and the LYME salt and drye. Therfore as soun as the Lye shalbe mingled with the Lyme, it must sone after nedis growe, so as the same be [due] in place mete for the purpose.
THE PLACIS mete for this mingling of the lye with the lyme, be QUARREES out of whiche, that kind of stone, which is called plaster is digged. So as the same be neither moyste nor subject to rayne or springe.
BARNES also standing of that heighte as there maybe five foote depthe digged in them.
MOREOVER VAULTIS in the cellars of monasteries and ruined houses of religion where the windowes may be shut and the storms (wind crossed out in manuscript) kept out, and be free from rayne and moisture: for it is specially to be loked unto that the houses be water tighte.
BUT IT IS BETTER to prepare some convenient place, wherin suche thinges as are herunto requisite may be [due] whiche is to be made as followeth.
TAKE NEW BRICK as it cometh out of the kiln, whiche never received rayne or other moisture, and let it be watered with the LYE wherof we have before spoken, and let it lye a sooking till it be throughly-moisted; and lay your bricke with the lyme, that is temperid with the said lye, and take the said dryed earth in steed of sande, and so make your wall, and let the same be crushid with the said morter or plaisster: and therby within a while, your wall shall bring forth as moche saltpeter as the earthe shall: for as it apearithe plainly, the saltpeter will hang upon the walles lyke snowe.

THE FLOWRE OR PAVIMENT must be made with the self same bricke and morter, that the walle is of.
AND THE LYME whiche is sonke downe to the botome of the killn, is to be taken out and raste upon sum flowre to drye, may be beaten small and mingled with the earthe, whiche by this means, will yeld as moche saltpeter, as th'earth itself for there is no cause why a good part of the lyme (that is to say the fourthe part) shuld, not be mingled withe the earthe, being (as it is said) the lyme itself, is no lesse frutefull, than the earthe whiche is strongeste for that it receiveth couldness out of the nature of the earthe.
THE PECIS OF THE LYME may be made as small as the thickness of a mans finger whereunto youe may put that rubbishe, whiche cometh of the breaking of the saide lyme.
THE EARTHE being brought to this point must be caste and settlid in the place appointed for this busines, three fote depe at the leaste, and muste be turned, and laboured like a garden grownde; but we must beware that we do not treade upon it, withe oure feete: and if it happen that we must passe over it, we must take some boorde to set under our feete.
AT FOURETENE DAYES END next after that the said earthe shalbe firste laboured, it must also be torned as men do tome wheate, so as the nether part must be tornid upwards, to th'intent th'earth may be made drye.
THE LYKE must also be done againe three wekes after, and then every moneth onis till that youe may perceive, that there is a good quantitite of saltpeter. THE WALLES AND VAULTIS must be swept often, for the Saltpeter will hang like snowe upon them.
TH'EARTHE being wrought in this sorte, muste be no more than twise stilled, to th'end that some part of the saltpeter, may still remayne; for if all of it shuld be drawne out, so as no remanente of the saltpeter shuld be lefte, it wold hardly growe againe, wherin he that shall meddle withall, had nede to be circumspect.
TH'EARTHE must be laide where it was, or nere the same place, and labored up newe, as it is saide before: and as often as it shalbecome drye, it must be moisted with the lye which is lefte, wherunto let some urine be added, stilled together with horsdong, wherin there muste be put a good quantite of lyme.
AFTER THAT one yeare is paste, youe may yearely stille this earthe twise: And to avoide the charge of wood, it may be boiled withe seacole.
BUT NOWE we must knowe, what ASHES we muste use, for without the same, the saltpeter cannot be made at all, whiche art I will willingly communicate to the Quenis mate, which is suche as requireth no charge, saving for workmanship; and maketh very good lye, and finally is made of suche stuff; as the ridding awaye therof ~vold be proufitable for the com¬mon wealthe, and wherof there is greate quantite in all place: and they be OKEN LEAVES, which are to be gathered in September, when they fall from the trees, and formed into ashes. And it proveth, that these ashes are very good, and that they be quicke and perc¬ing: for that where the Oken leavis do fall, and rot, no grasse dothe lightly growe, whiche ashes may be applyed to many other purposes.
HOWE the earthe is to be stilled and the saltpeter wrought out of it, is better knowne to every workman, than that it nedith here to be rehersid, yet I will say this moche further, that it is not good nor profitable that the saltpeter be boilde drye like salte, but that it be boilde with lye, as it is requisite: then let it be kept in cleane vesselles, that it may be like frosen ykles, for so shall it kepe it force still, whereas if it be boilde like salt, it loseth the viith part of his strengthe and goodnes. For so do the frenche men seeth it, whiche is not to be liked.
THE REMANENTIES of the lye, muste be diligently kept, to water the earthe, as often as it is formed, whiche watering muste be in a [wrane], as is used in the bleaching of lynen cloths in

the sonne.
URINE is fit for the making of the lye, albeit it be hard to come by.
IN those partes aforesaid, all the art is contained, and standeth, so as the same be handeled, as it oughte to be.

Notes

1 From: Williams A R 1975 'The production of saltpeter in the Middle Ages.' *Ambix* 22, 2, pp 125-33

2 Public Record Office, unpublished MS Ref. No. SP 12/16/29. Words in brackets are doubtful readings. The purchase of this recipe is mentioned in the Calendar of Patent Rolls of Elizabeth I 156 –63, II, 104; and a copy is noted in 1641 in the State Papers (Domestic) of Charles I, 487, 75. In 1578, Honrick described as a "native of West Friesland" applied for Letters Patent to give him, for 30 years "the sole right to erect certain engines invented by him for the draining of mines". Calendar of State Papers (Domestic), 125, 50. The saltpetre "hanging on the walls like snow" and the "ground turning white" (Turriano) may both be due to the same effect. My nitre-bed also acquired a white coating after a year or so, but this was not due to nitrates, or any salts, but a bacterial growth of some species.

Appendix 2

Suggested method for making saltpetre, taken from: Thomas Henshaw, 'The History of the Making of Salt-Peter and Gunpowder'. In Thomas Sprat, *The History of the Royal Society*, 1667

The manner of making saltpeter

- Take eight or ten tubs, each large enough to hold ten 'barrows' of earth. The tubs should have an open top, and fitted with a tap near the bottom.
- Place straw at the bottom of each tub, and on that some wooden boards which stop the earth from clogging up the tap hole.
- Arrange the tubs on their stands, and fill them with the nitrous earth to reach a hands breadth from the top of the tub.
- On the centre of the surface of the earth inside the tub, place a rundle of wicker of roughly 1ft diameter. Then next to this stick a 'good strong cudgel' into the earth. The wicker is to help the water spread evenly through the earth, and the cudgel when stirred allows the water to properly mix in with the earth.
- Pour cold water over the earth.
- Leave up to ten hours to settle, then let the water dribble (NOT run) through the taps. This provides the 'elixivium' or 'raw liquor'. Henshaw says if it the water does not run clear on the first instance, it should be poured on again and left to settle again, until the water produced is clear.
- Henshaw says you can test the strength of the liquor at this stage, by taking a glass jar of common water. Weigh the glass jar, then fill it with the lixivium. He says the difference in weight will indicate how much saltpetre is likely to be produced by this boiling.
- Over the same earth in the tubs, pour over more common water, to extract any remaining nitre. This liquor however will not be brought forward immediately to the boiling process however, but should be poured on new earth the next time, as it has some saltpetre in it.
- Then remove the 'useless insipid earth' from the tubs, which should be refilled in the same manner as before. Repeat until all the earth has been lixiviated.
- Take a copper tub, and fill with the liquor, and place on a brick work furnace.

- To one side of the furnace place a tub of the liquor which can dribble from a tap onto the copper at a speed suited to the fire quickly evaporating it. When boiled high enough, so that when a small part flicked onto a live coal, will 'flash like gunpowder'. Henshaw estimates this to be two days and one nights boiling, usually. At this point, 'a hundred weight' of liquor should contain about thirty five pounds of saltpetre. He claims the workmen usually just test this by noting that the liquor should behave like oil on the side of a knife or scummer.
- Next, two tubs should be prepared in the same way as done at the beginning with straw, boards, more straw (to stop the ashes floating to the top). Then fill the tub up with any kind of wood ash until about ½ foot from the top of the tub.
- Pour on the liquor hot from the copper on the ashes in the first tub, then after a while pour it off at the top. Continue pouring on and drawing off, alternating between both tubs of ashes, until the liquor is clear and without the thick colour it had when first poured on.
- At this point the greasy oil from the liquor has been trapped by the ashes. The liquor should be kept and be boiled again on its own. Whilst doing this, pour on hot water over the ashes, to drain out the remainder of the liquor.
- When starting the second boiling, first put into the copper the water that has just been run through the ashes, and as it heats, pour on the stronger liquor out of a tub as described earlier. Stand on the side of the furnace until the liquor in the copper is ready to crystallize.
- He notes that near the end of the boiling a lot of scum will emerge, which should be removed with a brass scummer with holes in it, and this should let some common salt fall to the bottom, which should be removed with the scummer also.
- You know the liquor is ready for this next stage when some of it dropped on a knife will coagulate like oil. He says at this stage every hundred pounds of saltpetre should have 'threescore and ten pound weight of peter'.
- Use iron ladles to transfer the liquor from the copper into a high narrow tub, called a settling tub. When the liquor is cool enough, common salt begins to stick to the sides of the tub. At the tap, placed about 1/2 foot from the bottom of the tub, draw out the liquor into deep wooden or brass trays, and leave them to shoot in a cool place. The cooler the better he says. The saltpetre produced will not be of good colour at this stage, but should be a mix between white, yellow and sometimes black.
- The salt at the sides of the settling tubs is common salt, which may be put to other uses.
- Let the liquor stand two days and two nights in the pans, and the liquor which swims on top of the saltpetre after this point should carefully be poured off.

To refine saltpeter

- Clean the copper, and add to it enough water as you think will dissolve the saltpetre to be refined. Boil the water and when hot, throw in the petre gradually, stirring with a ladle to help dissolve.
- Increase the fire until the liquor starts to boil. While doing this, use the scummer to check for and remove undissolved salt at the bottom of the tub, as this is common salt.
- As the water boils, scum the froth from the top.
- When boiled enough that a drop will coagulate, add a pint of strong wine vinegar or four ounces allom powder, and this will produce a black scum at the top. This should be allowed to thicken, so that it can easily be removed with the scummer.
- Lade the liquor into a settling tub, and cover with a cloth. After a couple of hours a thick yellow faeces will fall to the bottom. Then quickly draw off the liquor while hot, into shoot-

ing trays/pans, cover with cloth, and the liquor will start to crystalise. When no more will shoot, usually after two days, pour off the liquor into a tub with a hole at the bottom to train, then when try it is ready to use.
- The crystals should be sexangular, fistulous and hollow.

Equipment
- Nitrous earth
- Eight to ten large tubs with taps.
- Straw
- Wooden Boards
- Wicker rundles (1 foot diameter)
- Cudgel
- Copper pan/cauldron
- Brickwork furnace
- Wood/brass settling tubs/troughs
- Woodash
- Scummer (ladle with holes)
- 1 pint Wine vinegar

Timescale
- Day 1: prepare tubs of straw, boards and earth, and run through liquor.
- Day 1/2 (overnight): Leave tubs to settle.
- Day 2: Let tubs dribble through the taps; Rinse tubs through again
- Day 3: Fill copper with the liquor; boil for two days and one night.
- Day 4: Continue boiling in copper, fill tubs as before with any remaining earth.
- Day 5: Ash tubs to rinse boiled lixivium. Start second boiling of lixivium.
- Day 6/7: Transfer liquor to settling tub to crystallise.
- Day 8: Refining begins. Dissolve saltpetre in water, transfer to settling tub, and leave for about two days.
- Day 10/11: Saltpetre!