

REDISCOVERING PYROTARTARIC ACID: A CHEMICAL INTERPRETATION OF THE VOLATILE SALT OF TARTAR

Guido Panzarasa, Dipartimento di Scienze ed Innovazione Tecnologica (DiSIT), Università del Piemonte Orientale "Amedeo Avogadro," Alessandria, Italy, gp4779@gmail.com

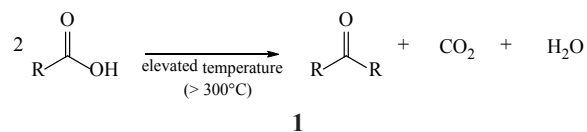
Abstract

Pyrolysis, the action of heat on organic substances in anaerobic conditions, has been one of the earliest not only analytical but also synthetic methods both in alchemy and chemistry. Applied to carboxylic acids this process allowed the preparation of a variety of chemicals, of which acetone is the best known example. But the nature of a product obtained from tartar, the *volatile salt of tartar*, generated a great deal of controversy among practitioners of alchemy because of its claimed involvement in the preparation of Paracelsus' *alkahest*. In this article the identification of volatile salt of tartar with pyrotartaric acid is suggested, by tracing the most relevant studies on the pyrolysis of tartar from alchemy to modern chemistry.

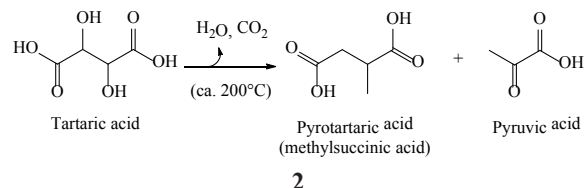
Introduction

The conversion of carboxylic acids to ketones, also called ketonic decarboxylation, is a useful chemical transformation that allows the production of symmetrical ketones. In this process, two carboxylic acid functionalities are converted into a carbonyl group with elimination of carbon dioxide and water (Scheme 1) (1). α - and β -ketoacids as well can undergo either thermal or enzymatic decarboxylation to yield ketones. Despite being one of the oldest reactions known in organic chemistry, it has very promising industrial applications because the

reactants are cheap, the by-products are innocuous and it doesn't require any solvent.



In this context, tartaric acid is a case of particular interest: subjected to pyrolysis, it gives two products, namely pyrotartaric acid and pyruvic acid (Scheme 2).



Although the history of the preparation of acetone by ketonic decarboxylation has been well documented since the first report in 1612 by Jean Beguin and is in fact common knowledge (2), that of pyrotartaric acid and pyruvic acid is not. This is rather confusing since the studies on the pyrolysis of tartar (the natural source of tartaric acid) and of the obtained products not only attracted the alchemists' attention earlier but were also taken into greater consideration because of their applications in medicine and in alchemical operations. Alchemists and Paracelsian iatrochemists claimed that it was possible to

extract a *volatile salt* from tartar, whose chemical nature remained controversial. The first detailed procedure for the preparation of this substance appeared in 1675 in Nicholas Lémery's *Cours de Chimie* (3).

The aim of the present article is to review the most relevant studies on the pyrolysis of tartar, starting from alchemy to modern organic chemistry, to suggest the identification of Lémery's volatile salt of tartar with pyrotartaric acid.

The Alchemy Period

Tartar is a saline concretion that wine naturally produces during storage in casks. The formation of tartar during vinification has been known since ancient times: it was initially called *faex vini*, and the word *tartarus* began to appear in the alchemical literature of the eleventh century (4, 5).

This body was the subject of intense research because the alchemists were attracted by its double nature, both vegetable and mineral. Tartar was early recognized as a valid source of useful chemicals: the first to be obtained were *cremor tartari*, which is tartar purified by crystallization from a saturated aqueous solution and *sal tartari*, an alkaline substance obtained by the calcination of tartar in open air. A variety of other derivatives have been described, such as the *tartarus emeticus* reported by Adrien von Mynsicht in 1631 (6) and the *Rochelle salt* prepared by Pierre Seignette around 1660 (7). Both were used in medicine, the first as an emetic and the latter as a purgative. Chemically speaking, *cremor tartari* is the monobasic potassium salt of tartaric acid or potassium bitartrate; by calcination in open air it is converted into potassium carbonate, which is a deliquescent alkaline solid melting at 891°C. *Tartarus emeticus* is an antimony potassium tartrate and *Rochelle salt* is a sodium potassium tartrate.

The uses of tartar spanned from pharmacy to metallurgy, where *fluxum nigrum*, obtained by partial calcination of tartar, was employed as a melting and (due to its high carbon content) reducing agent. By heating tartar with potassium nitrate—a process known as *detonation*—another kind of flux, *fluxum album*, could be obtained. *Fluxum nigrum* is a mixture of potassium carbonate and carbonized organic matter while *fluxum album* is potassium carbonate. The ability of tartar to increase the malleability of metals led to the belief that powerful agents for the transmutation of metals could be extracted from it. This is how the alchemist Johannes

de Monte-Snyder (~1625-1670) explained the effect of tartar on the melting of metals (8):

As the spirit of wine is known to be a sublime solvent and medicine, likewise tartar is not to be disregarded. Its spirit can be used for the unclogging of metals, thereby increasing their malleability and therefore also their salt; the malleability of metals depends in fact on the salt and any method by which they are made malleable acts by increasing the salt content. ... Tartar improves metals and makes them more malleable, joining them in the metallic salts formed from compounding with tartar. The malleability of metals resides in the abundance of salt, so if by the aid of Tartar a metal becomes more malleable at the same time the metallic salt must have increased. By means of Tartar the metals are changed into living Mercury, and this happens because while quantitatively the salt is increased, the Earth begins to predominate until the bond is broken. Indeed, when Earth increases, the Spirit which is not extensive but intensive is forced to expand excessively, causing dissociation and visibly contracting itself into quicksilver. [author's translation]

It should be noted here that according to the Paracelsian doctrine of *tria prima*, all bodies (including metals) are made of three substances or *principles* (*Mercury*, the principle of fusibility, volatility, malleability; *Sulfur*, the principle of color and combustibility; *Salt*, the principle of fixity) combined in different proportions.

In the words of de Monte-Snyder there is a strong echo of an alchemical treatise, *Duodecim Claves Philosophiae*, attributed to Basilus Valentinus (9):

In fact, the spirit of the salt of tartar, extracted per se, without additives, by solution and putrefaction, makes all metals volatile and reduces them to real living or fluent mercury, as is proven by my doctrines and practices. [author's translation]

Valentinus stated also that, if mixed with the spirit of common salt, this *spirit of tartar* can dissolve and volatilize silver and gold. It is important to state that in alchemy the term *spirit* was used to indicate volatile substances that could be sublimed and/or distilled. This led to a huge amount of work in order to obtain such a powerful solvent. Paracelsus (1493-1541) devised the word *alkahest* to describe a solvent capable of resolving a compound body into its constituent principles without being consumed or altered in the process (10).



Figure 1. From Valentinus' treatise *Duodecim Claves Philosophiae* (9): the twelfth key is an allegory of a metallurgical operation. Note the barrel on the left.

One of the most distinguished followers of Paracelsus, Jean Baptiste van Helmont (1579-1644), stated the possibility offered by the volatilization of the salt of tartar to obtain a powerful solvent, although not as universal as the alkahest (10):

If you cannot attain this arcanum of fire [i.e., the alkahest], learn then to make salt of tartar volatile and complete your dissolutions by means of it.

Following this claim an incredible number of procedures to obtain such a volatile salt of tartar were proposed, which can be divided basically into two categories. The first ones were based on the controlled action of heat on tartar in accordance with the precepts of Valentinus, while the second ones tried to induce volatility in the salt of tartar by repeated distillation with volatile spirits like spirit of wine, spirit of vinegar, sal ammoniac (10). To this latter category belongs the efforts of iatrochemists like Franciscus Sylvius (1614-1672) (11) and George Starkey (1628-1665) (12) who both obtained relevant results in studying the interaction of *sal tartari* with essential oils. In such a climate of passionate and disordered research, as the alchemist Blaise De Vigenère (1523-1596) wrote, tartar became "one of the subjects which those who experiment with fire greatly abuse" (13).

Nicholas Lémery's Volatile Salt of Tartar

In 1675 the iatrochemist Nicolas Lémery (1645-1715) published his famous *Cours de chimie*, the first attempt to make the operation of chemistry and especially the preparation of medicinal compounds easy to understand and to execute. His intention was to describe each operation first as a simple recipe and only afterwards

present a mechanical explanation for it. Lémery was the first not only to report a detailed description of the action of heat and fire on tartar but also to clearly state the extraction of a *volatile salt* from tartar by means of dry distillation (14):

This Operation is the Salt of the Lees of Wine, volatilized by Fermentation.

Dry the Lees of Wine with a gentle Fire, and fill with them two thirds of a large earthen or glass Retort, place this Retort in a Reverberatory Furnace [(15)], and fitting to it a large Receiver, give a small Fire to it to heat the Retort by Degrees, and to drive forth an insipid Phlegm, and luting carefully the Junctures of your Vessels, quicken the Fire by little and little, until you find the Receiver filled with white Clouds; continue it in this Condition, and when you perceive the Receiver to cool, raise the Fire to the utmost Extremity, and continue it so until there rise no more Vapours. When the Vessels are cold, unlute the Receiver, and shaking it about to make the volatile Salt which sticks to it fall to the Bottom, pour it all into a Bolt-head [that is, a distillation flask], fit to it a [distillation] Head with a small Receiver; lute well the Junctures, and placing it in Sand, give a little Fire under it, and the volatile Salt will rise, and stick to the [distillation] Head and the Top of the Bolt-head; take off your [distillation] Head, and set on another in its Place: gather your Salt and stop it up quickly, for it easily dissolves into a Liquor; continue the Fire, and take Care to gather the Salt according as you see it appear; but when there rises no more Salt, a Liquor will distill, of which you must draw about three Ounces and then put out the Fire.

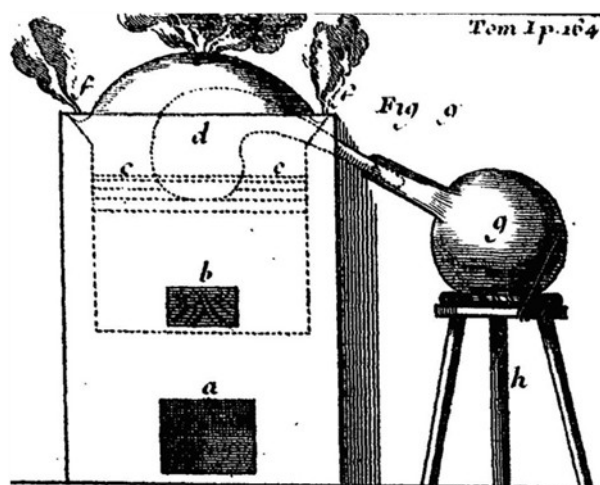


Figure 2. Reverberatory furnace for pyrolysis experiments (16).

Lémery described three principal products from the distillation of tartar: a *tasteless phlegm*, which in

the alchemical speech indicates a watery distillation, a white *volatile salt* and an unidentified *liquor*. In another work on this subject he reacted *cremor tartari* with the alkaline *sal tartari* obtaining *soluble tartar* (a neutral potassium tartrate), in this way demonstrating the presence in tartar of an acid. Nevertheless, a clear distinction between the acid of vinegar and the acid of tartar had not been made until the first decades of the 1700s. Even the great iatrochemist Hermann Boerhaave (1668-1738) considered this two acids to be identical; otherwise he would not have considered potassium acetate, obtained by saturating distilled vinegar with *sal tartari*, as *regenerated tartar* (17). The existence in tartar of a peculiar acid different from acetic acid was definitely recognized by Carl Wilhelm Scheele (1742-1786) in 1769, who isolated tartaric acid.

More than a century after Lémery, the chemist Pierre-Joseph Macquer (1718-1784), published another description of the analysis of tartar by pyrolysis (18):

Tartar analyzed by distillation. The Spirit, Oil, and Alkaline Salt of Tartar.

Into a stone retort, or a glass one coated with lute, put some white Tartar broken into small bits; observing that one half, or at least a full third, of the vessel be empty. Set your retort in a reverberating furnace. Fit on a large ballon, having a small hole drilled in it; lute it exactly with fat lute, and secure the joint with a linen cloth smeared with lute made of quick-lime and the white of an egg. Apply at first an exceeding gentle heat, which will raise a limpid, sourish, pungent water, having but little smell, and a bitterish taste.

When this first phlegm ceases to come off, increase your fire a little, and make the degree of heat nearly equal to that of boiling water. A thin, limpid Oil will rise, accompanied with white vapours, and with a prodigious quantity of air, which will issue out with such impetuosity, that if you do not open the little hole in the receiver time enough to give it vent, it will burst the vessels with explosion. An acid liquor will rise at the same time. Continue the distillation, increasing the heat by insensible degrees, and frequently unstopping the little hole of the receiver, till the elastic vapours cease to issue, and the oil to distill.

Then raise your fire more boldly. The acid Spirit will continue to rise, and will be accompanied with a black, fetid, empyreumatic, ponderous, and very thick Oil. Urge the fire to the utmost extremity, so that the retort may be of a perfect red heat. This violent fire will raise a little Volatile Alkali, besides a portion of Oil as thick as pitch. When the distillation is finished, you will find in the retort a black, saline, charred matter, which grows hot when wetted, attracts the

moisture of the air, runs *per deliquium*, and hath all the properties of a Fixed Alkali.

The mass, being exposed to a naked fire in the open air, burns, consumes, and is reduced to a white ash, which is a fiery, caustic, Fixed Alkali.

In accordance with the ruling concepts of his day, Macquer (erroneously) thought that the only result of dry distillation was the separation of the components of a mixture. He compared the results obtained from the pyrolysis of tartar, of the lees of wine and of the residue obtained by distillation of wine concluding (p 272) that

...liquors, which have undergone the spirituous fermentation, consist of an Ardent Spirit and a Tartarous Acid suspended in a certain quantity of water.

Pyrolysis was considered a valid method for the study of organic acids and of their derivatives, typically metallic salts. The prefix *pyro-* was assigned to the substances obtained by the action of heat without the addition of chemical reagents. *Pyro-acetic acid spirit* was obtained from acetates and in 1833 renamed as *acetone* (2) while the principal product obtained by the pyrolysis of tartaric acid was named *pyrotartaric acid*. A classification of organic acids into *volatiles* and *non-volatiles* was then proposed (19) on the basis of their behavior on being subjected to strong heat. It is interesting to note that such a distinction, although with a slightly different meaning, still has use in physiology, to indicate the kind of acids typically produced by the human body, and in botanical sciences. (Non-volatile acids are those that originate from the metabolic transformation of proteins contained in food, while the only volatile acid is carbonic acid.)

However, despite centuries of practice, dry distillation was still in its infancy (20):

The products resulting from the dry distillation of organic substances have recently been the subject of particularly thorough study. ... Despite the fairly important findings that have been made, it is evident that this subject is still in its cradle and that the so-called dry distillation of organic substances opens up an entirely new field in chemistry. ... The same substances—or mixtures of substances—may give rise to different products, according to temperature and the admixture of different inorganic substances.

By the involvement of skilled chemists, a rigorous chemical investigation of tartar could then start. Dedicated investigations of pyrotartaric acid were made by Théophile-Jules Pelouze (1807-1867), who believed that “the action of heat on tartaric acid represents one of the most interesting subjects from the history of pyrolyzed bodies” (21). According to Thomson (19, pp 63-64):

This acid [pyrotartaric acid] was made known to chemists in 1834. M. Pelouze obtained it by exposing tartaric acid to heat in a retort. (Ann. de Chim. et de Phys. lvi. 297)

When tartaric acid in a retort is kept at the temperature of 374° [Fahrenheit], a liquid passes into the receiver, which yields, when evaporated, pure crystals of *pyrotartaric acid*. But this process is so very tedious, that is better to distil at a temperature between 392° and 572°. The liquid that passes over is to be put into a retort and distilled to the consistence of a syrup. We must then change the receiver, and continue the distillation to dryness. The pyrotartaric acid is contained in this last liquid. When we surround it with a freezing mixture of snow and salt, or leave it to evaporate spontaneously, *in vacuo*, over sulphuric acid, yellow crystals are deposited. Dry these between folds of blotting paper, dissolve them in water, and agitate the boiling solution with animal charcoal, then crystallize. The crystals constitute pyrotartaric acid in a state of purity. (This acid may be obtained also by distilling cream of tartar in a retort. See the experiments of the Athenian, Alexandros Polaiologu Weniselos, in Ann. der Pharm. xv. 147.)

Pyrotartaric acid, thus obtained is in white crystals, which have an exceedingly sour taste, but no smell; and are very soluble in water and alcohol. When heated to 212° the acid melts, and it boils at 370°. This being very near the point of decomposition, it is difficult to volatilize it without residue. ... When racemic acid is distilled, it yields an acid identical with pyrotartaric acid.

Pelouze observed also that when tartaric or racemic acid is distilled in a retort, another acid besides pyrotartaric acid forms, which he considered to be acetic acid. (From the Latin *racemus*, "grape," racemic acid is a 1:1 mixture of D- and L-tartaric acid.) Justus von Liebig (1803-1873) first distinguished it from acetic acid and regarded it as a *liquid pyrotartaric acid* (22):

Liquid pyrotartaric acid. To prepare it, one subjects crystallized tartaric acid to a temperature of 200°C in a large tubulated retort with the aid of a spirit burner. The mass melts and soon swells considerably. ... Eventually, a heavy acidic liquid, with a strong smell of vinegar, distils and at the same time carbonic acid is liberated. When the distilled product begins being colored, the distillation is stopped. The residue in the retort is syrupy, dark-black colored, insoluble in water, soluble in water [*sic*; should be alcohol, as in the original German] and in alkali. ...

Solid pyrotartaric acid. This acid forms with low yield during the distillation of tartaric acid. It is obtained with higher yields by distilling the acidic potassium tartrate (Weniselos). To prepare it, a glass

retort is two-thirds filled with cream of tartar, which is then distilled; the colored acid obtained is subjected to evaporation in a water bath until crystallization starts; the solid pyrotartaric acid separates on cooling.

Jöns Jacob Berzelius (1779-1848) examined this liquid pyrotartaric acid in 1835 and eventually recognized it as a completely new substance (20):

... I pointed out that racemic acid on dry distillation produces an acid different from the pyrotartaric acid arising from tartaric acid, which has been well known for a long time. ... Although I have confirmed the findings of Pelouze that tartaric acid and racemic acid give the same distillation products, I did not reach the same conclusion that he did concerning the nature of the main part of the distillate, which he considered, because of its smell, to consist of acetic acid. This acid ... I called pyruvic acid and described as a volatile acid. I will go on using the name pyruvic acid, *acidum pyruvicum*, as an indication of its origin from the grape. The same acid is also found in the acidic fluid which is formed when solid potassium tartrate is submitted to dry distillation; it consequently occurs in an ancient remedy, *mixtura diatrion* or *m. pyrotartarica* [(23)] and deserves to be known for this reason as well.

The description made by Berzelius is a very accurate one (20):

I distilled a sample of tartaric acid in a tubulated glass retort on a sand bath, the temperature of which could be kept almost constant by increasing or reducing the draught. The mass started to give off a water clear distillate of a sharp taste, smelling of acetic acid, whereupon it melted, turned grayish, and began to foam.

When the material showed a tendency to boil over, the cork was removed from the tubular extension and a rigid platinum wire was introduced to break the foaming, which was easily accomplished. It was sometimes necessary to repeat this procedure. Without this precaution the distillation proceeds extremely slowly and the entire foaming mass collects in the upper part of the retort leaving the lower part empty. During the operation carbon dioxide is developed in a continuous stream, necessitating tubulation of the receiver, the end of the tube being submerged in water.

The content of the retort gets very sticky before the end of the operation, mostly due to condensed pyruvic acid. Later on in the operation the distillate gets more and more concentrated and becomes yellow in color, but even at the end is no more than pale yellow if the temperature does not exceed +220° (Celsius [*sic*]). The residue in the retort becomes more and more sticky, finally changing to a fluid which boils without foaming; the temperature is then about +200°, and

when the boiling is finished and nothing more distills at +200° the operation is finished. There is then left in the retort a mass which is soft while still hot but after cooling behaves like carbon.

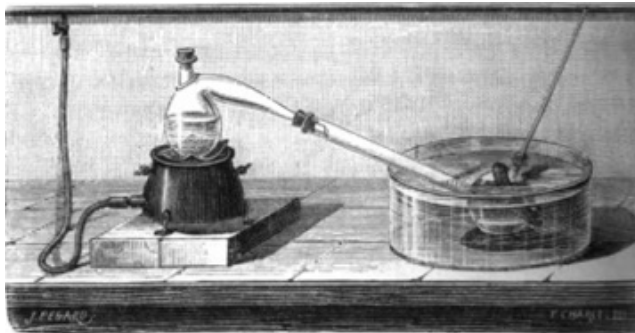


Figure 3. Eighteenth century distillation apparatus: retort and tubulated flask (24).

Berzelius was able to isolate pyruvic acid in a pure form by decomposing the lead salt with hydrogen sulfide. He was unable to obtain pure pyruvic acid by means of distillation alone because of its decomposition during heating (20).

Berzelius was a convinced promoter of the radical theory of organic compounds and he ended on considering pyruvic acid to be “a lower degree of oxidation of the same radical” as that of tartaric acid. In addition, on the basis of their formulae Berzelius found formic acid, tartaric acid, pyrogallic acid and pyruvic acid to be derived from the same organic radical.

Modern Organic Chemistry and the Solution of the Enigma

Several products can be obtained by heating tartaric acid at different temperatures. Two key processes are involved in their formation, namely dehydration (i.e.

loss of water) and decarboxylation (i.e. loss of carbon dioxide) (25). A prolonged heating at 150°C brings about the elimination of water from tartaric acid and the formation of tartaric anhydride, a white powder which readily recombines with water to give tartaric acid (26).

If the temperature is raised to 200°C decarboxylation of tartaric acid takes place and carbon dioxide is evolved while pyrotartaric acid is formed. At the same time, both tartaric acid and pyrotartaric acid decompose into pyruvic acid which in turn decomposes slightly into acetic acid by decarboxylation. Minor products are carbon monoxide, formic acid, acetoacetic acid and ethylene. It is evident that the nature and amount of these products is strongly dependent on the temperature at which pyrolysis is performed (27). Figure 4 summarizes the key steps of this process.

It is now possible to conclude that the product described by Nicholas

Lémery as volatile salt of tartar, which appears as white clouds condensing in a solid when the tartar of wine is subjected to pyrolysis, is pyrotartaric acid also known as methylsuccinic acid. The formation of this product is typically followed by that of an *acid liquor* which is a mixture of pyruvic acid and acetic acid.

The *tasteless phlegm* is the water produced

by the dehydration of tartaric acid while the *huge amount of air* that impressed Macquer is carbon dioxide, generated by decarboxylation.

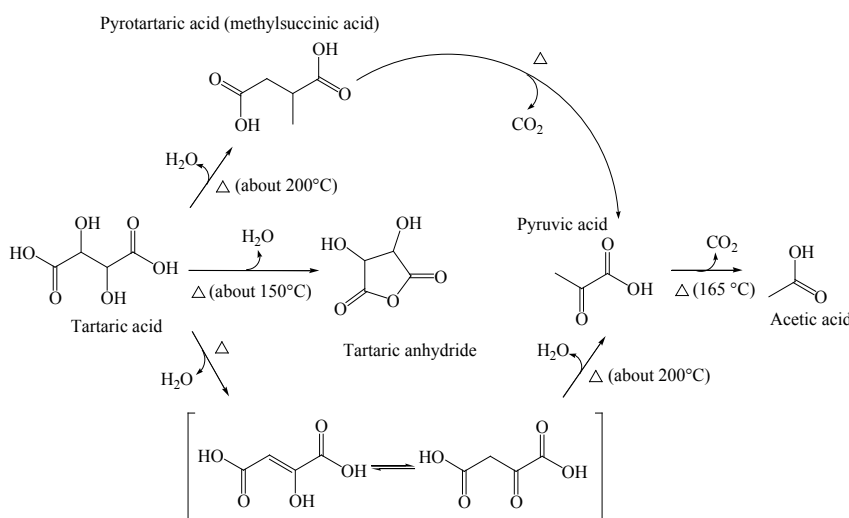


Figure 4. The principal processes involved in the pyrolysis of tartaric acid and the obtainable products.

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15. Reverberatory furnaces were used when high temperatures were required and were typically made of refractory bricks welded with a clay paste. The dimensions of these kilns varied depending on the size of the retorts to be employed. Retorts were supported by two iron bars placed inside directly on the fireplace, whose dimensions were such as to allow an easy ignition and maintenance of the fire. Under the fireplace were the air intake and the ashtray. The heat had to surround the retort completely to prevent the condensation of the vapors evolved from the processed substance, so the kiln was covered with a dome equipped with an exhaust vent for the fumes.
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About the Author

Guido Panzarasa, born in 1988, graduated from the Università del Piemonte Orientale "Amedeo Avogadro" in Materials Science (B.Sc., 2010) and in Chemical Sciences (M.Sc., 2012). He will obtain his PhD in Chemical

Sciences at the same Institution in 2015, working in the field of polymer chemistry and nanotechnology, in particular polymer brushes. His research interests span from the development of innovative patterning strategies (e.g. photocatalytic lithography) to advanced characterization techniques (e.g. positron annihilation spectroscopy). His interest in the history of chemistry is focused on the analysis of ancient procedures and concepts with a contemporary chemistry approach.

HIST Award 2015

The recipient of the 2015 HIST Award of the Division of the History of Chemistry of the American Chemical Society is Professor Dr. Christoph Meinel of the University of Regensburg, Germany. This award is the successor to the Dexter Award (1956-2001) and the Sydney M. Edelstein Award (2002-2009), also administered by the Division of the History of Chemistry (HIST) of the American Chemical Society.

The HIST Award consists of an engraved plaque and a check. It was presented to Professor Meinel at the fall national meeting of the American Chemical Society in Boston in August 2015. Additional information about Prof. Meinel, the award, its past recipients, and its past incarnations can be found on the HIST website at http://www.scs.illinois.edu/~mainzv/HIST/awards/hist_award.php.

