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Reflexiones

Charles Cagniard de Latour

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Resumen

Charles Cagniard de la Tour (1777-1859) fue un físico y químico francés que realizó un estudio detallado de la transformación de fase de un líquido saturado bajo la influencia de la presión y la temperatura y demostró que dentro de un tubo de vidrio sellado el fluido se convertía primero en un vapor saturado, y luego continuamente en vapor sobrecalentado, sin importar la presión. Descubrió el fenómeno del punto crítico, sin usar este término. Cagniard de la Tour usó un microscopio para investigar el fenómeno de la fermentación, particularmente por la levadura de cerveza, sus causas y efectos, y demostró que era causado por un organismo vivo y no era una reacción química pura. También estudió la pirolisis de materia leñosa en una atmósfera sellada y demostró que resultaba en la formación de material carbonoso alquitranado y pegajoso, acompañado de una atmósfera de gas.

Palabras clave

Alquitrán; fermentación; pirolisis de la madera; punto crítico.

Abstract

Charles Cagniard de la Tour (1777-1859) was a French physicist and chemist that carried a detailed study of the phase transformation of a saturated liquid under the influence of pressure and temperature and proved that inside a sealed glass tube the fluid would become first a saturated vapor, and then turn continuously into superheated vapor, no matter what the pressure. He discovered the existence of a critical point, without using this concept. Cagniard de la Tour used a microscope to investigate the phenomenon of fermentation, particularly by beer yeast, its causes, and effects, and proved that it was caused by a living organism and was not a pure chemical reaction. He also studied the pyrolysis of woody matter in a sealed atmosphere and showed that it resulted in the formation of tarry and sticky carbonaceous material, accompanied by a gas atmosphere.

Keywords

Tar; fermentation; wood pyrolysis; critical point..

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Life and career

FIGURE 1. Charles Cagniard de Latour (1777-1859).

Little information is available about the early life and career of Charles Cagniard de la Tour (Figure 1). He was born in Paris, on March 31, 1777. The normal course of his higher studies was affected by the events of the French Revolution. He started them at the École Royale Militaire de Rebais, created under the King Louis XVI and closed in 1793 by the French Revolution. together with many academic and educational institutions. In 1794, the mathematician Gaspard Monge (1746-1818), then Minister of the Marine, replaced the École de Rebais by the École Polytechnique, which was afterwards (1804) converted into a military engineering academy by Napoleon Bonaparte (1769-1821). Cagniard de la Tour completed his studies in the first graduation of the new school, as *ingénieur géographique* (engineering geographer, topographer), and from there, in 1811, he went to work at Ministry of the Interior. In 1818 he was made a baron after successfully supplying the Hôpital Saint Louis in Paris and the Usine Royale d'Armes in Saint-Etienne, with gas lighting.

Cagniard de la Tour carried a successful research career that led him to the invention of a blowing machine, a siren for determining the number of vibrations corresponding to the sound of any pitch, and basic subjects as sound and the mechanism of human voice, the effect of pressure and temperature upon constant volume systems and the causes and effects of fermentation. The work on heating and pressure led him to postulate the presence of a critical state for every pure compound, although he did not use this expression. Heating a limited amount of saturated liquid in a sealed glass tube led to all the tube becoming full of saturated liquid or saturated vapor, depending on the initial density being smaller or larger than the critical one. For each fluid, there was a temperature above which, it passed completely first into the saturated gaseous state and then into superheated vapor, no matter what the amount of pressure to which it was subjected. Independently of Theodor Schwann (1810 1882), he postulated that fermentation was produced by a living organism and not by a pure chemical reaction, and that yeast could support very low temperatures and absence of water.

Cagniard de la Tour was auditor of the Conseil d'État (Board of State) and member of the Société d'Encouragement de l'Industrie; in 1850 he was elected member of the Académie des Sciences (section Physique) replacing Joseph-Louis Gay-Lussac (1778-1850) and defeating Léon Foucault (1819-1868) by 34 votes against 19.

Cagniard de la Tour died in Paris on July 5, 1859.

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Scientific contribution

Cagniard de la Tour wrote more than 45 papers and books (i.e., (Cagniard de la Tour, 1833, 1834; Cagniard de la Tour & Turpin, 1835) about the physics of sound and voice, thermodynamics, inorganic chemistry, physiology, etc. As customary for candidates to the Académie des Sciences, he published a booklet describing the results of his scientific research (Cagniard de la Tour, 1851a). In addition to the subjects described below, he designed a new dynamometric apparatus to measure the dynamic effects of operating machinery (Cagniard de la Tour, 1837a); analyzed the mechanical properties of Dutch drops and suggested and explanation for them (Cagniard de la Tour, 1837d); developed a process for synthesizing small diamonds (Cagniard de la Tour, 1847); etc. Cagniard de la Tour did also significant research about physical phenomena, particularly, vibration sound, and the human voice (i.e., Cagniard de la Tour, 1809, 1819, 1830, 1833, 1834a, 1836bd, 1837e, 1840, 1851b). These will not be presented here [part of these studies led to the development of practical instruments (i.e., Cagniard de la Tour, 1809,1819)].

Critical point

According to Cagniard de la Tour, it was well known that the temperature a liquid could be raised about its boiling point at atmospheric pressure, by heating it in a Papin pot (a pressure cooker); and it could also be anticipated that the internal pressure would increase with the temperature and become an obstacle to the total conversion of the liquid into vapour, especially when the space left above the liquid was limited. Despite the increase in pressure, the expansion of a volatile liquid had an upper limit, beyond which the whole liquid phase would be converted into vapor (Cagniard de la Tour, 1822ab).

For a better understanding of these and the following arguments and experiments of Cagniard la Tour, it is interesting to consider what Pierre Duhem (1816-1916) wrote more than seventy year later (Figure 2) (Duhem, 1894).

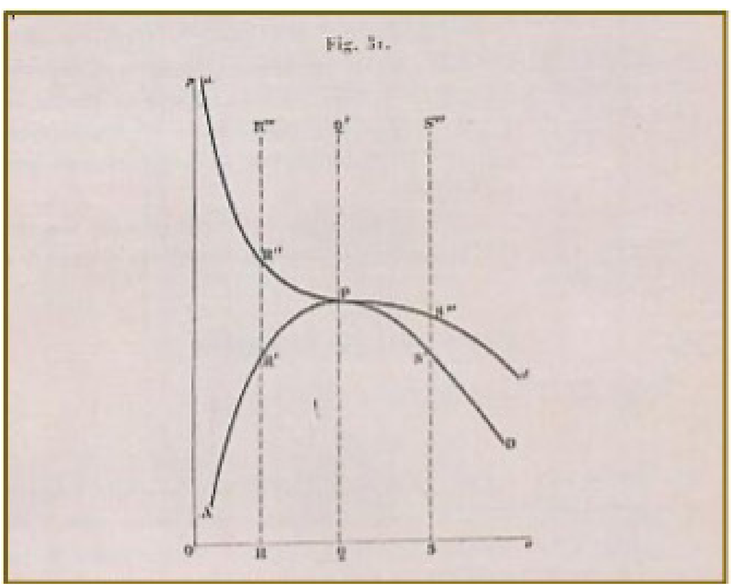

Figure 2. Pressure-volume phase diagram of a pure substance (Duhem, 1894).

The curve *dd* represents the critical isotherm, *P* the critical point, and the curve *ADP* the saturation curve, where *AR'*P is the specific volume of the saturated liquid and *PS'D* that of the saturated vapor. Any point below this curve represents a liquid in equilibrium with it vapor. In the case of a constant volume system, like a closed tube, its behavior is represented by a vertical curve such as *RR'* and *SS'*. Two cases can be distinguished depending on if the vertical curve is located to the right or left of point *P*, that is, the combined density of the

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liquid and vapor phases is smaller or larger than the critical density. In the first case (line *SS'*), heating the tube increases the amount of vapor present in the tube until all the liquid disappears upon reaching the state *S"* (saturated vapor).

Further heating turns the contents into superheated vapor until reaching point *S*' where it becomes a superheated gas, without discontinuity. In the second case, heating causes that amount of liquid to increase until it reaches point *R*' where the tube is full of liquid, If the initial density is that represented by point 2, heating the tube will lead to the critical point. In other words, proper selection of the initial amount of liquid in the tube will result in an increase or decrease of density of the fluid mixture with the temperature. The critical point is the interface between both processes (Duhem, 1894).

All this background was not available at the time of Cagniard de la Tour, making his conclusions so more significant.

In the first stage, he filled a piece of a strong iron gun barrel with about two-thirds of alcohol and added a small ball of silex to serve as an indicator of the changes in internal density by the noise it produced noise when rolling the barrel backward and forward. The barrel was then sealed and heated over burning charcoal, while moving it constantly. Cagniard de la Tour noted that the noise became more audible as the temperature increase, suggesting that less liquid was present. The noise decreased when the contents were cooled. The same results were observed when diethyl ether of or rectified petroleum oil replaced the alcohol. This was not the case with water because the sealing failed with the high pressure developed by the steam. (Cagniard de la Tour, 1822ab).

These experiments were repeated with the same liquids contained in small glass tubes, closed at both ends by the flame of a blowpipe. This arrangement allowed a visual observation of the changes that were taking place. Cagniard de la Tour remarked that all these tubes were emptied of air before being sealed. Thus, one of these tubes, filled with alcohol to about two-fifths of its capacity, showed the slow disappearance of the liquid phase until the contents seemed to be completely made of a transparent vapor, which reappeared a thick cloud upon cooling. Further cooling was formed, caused the complete recovery of the original liquid phase. Another tube filled with a larger amount of liquid exploded before achieving total vaporization. All the liquids tested produced similar results, even when air was left in their insides. Cagniard de la Tour asked himself if the liquids, which were capable of but a small degree of compression at a low temperature, become more compressible at a higher temperature, and much more so in the present case, when the liquid was on the point of being converted into an elastic fluid under a pressure amounting probably to several hundred atmospheres (Cagniard de la Tour, 1822ab)?

Cagniard de la Tour went one step further and tried to determine the pressure and temperature at which ether and alcohol achieved complete vaporization (Cagniard de la Tour, 1823a). For this purpose, he used an apparatus constructed like a siphon barometer, that is, of a tube of 4.5 mm internal diameter, bent like a hook and welded to a longer leg of internal diameter 1 mm and the most equal caliber that was possible to find. After the mercury and the ether were introduced then the two open ends were sealed. The dimensions were such that 1 mm of variation in the larger tube produced a variation of 20 mm in the small one. The columns were then property graduated. The apparatus was heated over an oil bath and measurements taken up to the point when the ether was completely evaporated (pointed S, in Figure 2). The results indicated that the pressure and

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the temperature at this stage was between 37 and 38 atmospheres and 200 °C, respectively (average of three experiments). For alcohol of 36° (not pure), they were 119 atmospheres and 258.75 °C. Cagniard de la Tour provided a table describing for ether and carbon disulfide the temperature and pressure at several intermediate stages (Cagniard de la Tour, 1823a). Today we know that the critical pressure and critical temperature for ether and carbon disulfide are (35.53 atm and 194 °C) and (77.97 atm and 279 °C), respectively. Cagniard de la Tour wrote that in another publication he would provide a drawing of his experimental apparatus (Cagniard de la Tour, 1823a). It seems that he never did.

Another short note reported the results of similar experiments conducted on binary and ternary systems (Cagniard de la Tour, 1823b). Water was introduced in a glass tube together with a small amount of carbon disulfide (relative density 1.26 at room temperature). Upon heating, the water turned first milky, then recovered its transparency with a slight tint of green, and finally became almost black. The carbon disulfide, initially at the bottom of the water phase became lighter than the water and floated on it sometime before it became vaporized completely. The tube exploded before the water phase became totally vaporized. Another experiment was stopped before the explosion and the tube left to cool. The process now reversed: the green color diminished by degrees, and the carbon disulfide became heavier than water while the latter assumed a yellowish color (probably partial decomposition of the sulfide) (Cagniard de la Tour, 1823b).

Another tube contained water, a little carbon disulfide, and a little potassium chlorate. The initial heating caused the salt to dissolve and the carbon disulfide to become lighter than water. Letting the liquid to cool resulted in the water becoming milk and the carbon disulfide to return to the bottom accompanied by crystallization of the potassium chlorate. Repeating the experiment while heating to a higher temperature, and without cooling, led the carbon disulfide to float, the liquid to turn lemon yellow, accompanied by effervescence, and the formation of an oily looking globule which, when all was cold, remained, liquid at the bottom of the tube, with no deposition of crystals (Cagniard de la Tour, 1823b).

A new confervoid micro plant

At some time, Cagniard de la Tour became interested in what happened when ordinary drinking water was placed in contact for a long time with a mixture of air and acetic acid vapor (Cagniard de la Tour, 1835). For this purpose, he placed in his cellar a stemmed glass filled with filtered water of the Seine and immersed in it the open end of a tube curved into a siphon, the closed branch of which contained about two grams of wood vinegar in contact with atmospheric air. After same time, Cagniard de la Tours noticed that the mixture of air and acetic acid vapor that had dissolved in the water had led to the development of a white fluffy material, which increased in amount with time and turned dark green almost black, a coloration that was remarkable as the cellar was in almost complete darkness (Cagniard de la Tour, 1835).

After about eight months, Cagniard de la Tour had the opportunity of showing this material to the botanist Pierre Jean François Turpin (1775-1840). Turpin examined it under the microscope and reported that it was composed of very tight filaments, confervoid, without partitions, colorless, and armed with pointed branches like a thorny plant. All these characteristics pointed out that Cagniard's material formed an altogether new species.

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A second arrangement was found after three months to show already several small clusters of a plant production like the previous one, which suggested that the same process was accessible in any liquid (or the air in contact with it) that contained the *seminules* (corpuscles) suitable for reproducing the species of conferve. Cagniard de la Tour moved the device from the cellar to a room at 10 °C and in diffused light and noticed that under these conditions the growth process became more active. This result led him to repeat the experiment putting a fresh arrangement directly in a lighted room at ordinary temperature: the growth took place as before except that this time the fluffy material was almost colorless. The same experiment conducted in the absence of acetic acid, hardly gave place to the formation of vegetable material.

In another experiment, Cagniard de la Tour placed two fresh devices in his cellar, one containing rainwater, and the other water drawn from a well. Under the action of the air mixed with acetic acid vapor, both liquids became cloudy after a few days, as had happened with the water from the Seine. Filamentous flakes were now seen in both glasses, but the rainwater product was more abundant than that of well water.

Cagniard de la Tour tested from time to time with litmus paper the water in his glasses and noticed that with the devices placed in the cellar the water had an acid reaction, which usually manifested itself fifteen days after the experiment had started. This acid reaction disappeared sometime after the glass had been transferred to the room in diffuse light and at 10 °C and did not take place when the experiment was started directly in this room.

According to Cagniard de la Tour, the absence of acidity in these last two devices was undoubtedly since the water evaporated more quickly, and by this means deposited enough calcium carbonate to neutralize the acid (acetic) as it was supplied by the air from the siphon.

Cagniard de la Tour mentioned that Henri Dutrochet (1776-1847) had found that a very small dose of HCl or other acid added to an aqueous solution of egg albumin, promoted the development of molds in this solution (Dutrochet, 1834) and that Henry Milne Edwards (1800-1885) and Jean-Jacques Colin (1784-1865) had reported that the germination of seeds of cereals on slightly acid water was accompanied by mold growth (Edwards and Colin, 1836; Cagniard de la Tour, 1835). Cagniard's paper was followed by another by Turpin giving a detailed microscopic description (as well as their drawing) of the conferves discovered by Cagniard de la Tour (Turpin, 1835).

Siliceous and calcareous materials obtained by slow reactions

In 1837 Cagniard de Latour published a paper announcing that he had developed several processes to synthesize, by slow reactions, analogues of naturally occurring substances, such as diamonds, calcium-based feldspar, and crystalline calcium carbonate (Cagniard de la Tour, 1837b). For example, carbon black was heated with concentrated nitric acid, and the liquor after having been decanted, was exposed under a glass bell, for several months to the action of sunlight, replacing the eliminated acid or water, as required. Little by little siliceous concretions formed, some of which assumed the pyramidal shape and contained 2% carbon. Treatment of these concretions with hot KOH in a platinum crucible diminished their size and left as residue a material hard enough to scratch rock crystal (Cagniard de la Tour, 1837b).

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Fermentation

Cagniard de la Tour carried extensive research about the causes and effects of fermentation, and about its nature (Cagniard de la Tour, 1836ac, 1837b, 1837ac, 1838a; Cagniard de la Tour and Turpin, 1838). He wrote that in 1800, the class of physical and mathematical sciences of the Académie des Sciences had proposed, as subject of a prize, the following question: *What are the characteristics which distinguish, in plant and animal matters, those which serve as ferment from those for which they undergo fermentation*? Although this was a general question, its main purpose was the conversion of sugar into alcohol and CO_2, that is, the *vinous fermentation*. It was well-known that mixing a sugar solution with fresh brewer's yeast at about 25 °C, the solution began to ferment, rapidly according to the amount of yeast. This process did not take place in the absence of yeast or if the sugar of the solution was not pure. No one won the prize. It was offered again in 1802 and was withdrawn in 1804 after an event that left the Académie without the funds to finance it (Cagniard, 1838a).

Cagniard de la Tour considered the question still valid and decided to study it differently than had been done before, this time, using a microscope, based on what he had learned from previous experiments in which he had studied with the aid of a microscope, the main phases of the fermentation taking place in the wort of beer after it had been placed in leaven (Cagniard de la Tour, 1836b). He had noticed that soon the must appeared to contain a considerable number of very fine particles, but without definite forms. Sometime later, several of the particles were double, each had a secondary or smaller globule, which seemed to have grown as if by extension of the main globule. After the foam formed by the fermentation had achieved its ordinary maximum height, it was possible to observe globules united three to three, four to four, sometimes even in greater number, and usually forming fragments of strings; it was further noted that the number of globules was obviously much greater than in the first sample taken out after setting in leaven. A few days later, the flask contained nearly seven times the weight of the leaven used; curiously, this time most of the globules were single, which would indicate that as they got older, they became detached from each other. This subsequent disunity, which could scarcely be attributed to anything other than a vital action, removed the idea that the formation of globules could be considered as a pure effect of crystallization or albuminous coagulation. Cagniard de la Tour mentioned that while the single globules had sharply drawn outlines, the multiple ones were seen as through a cloud. He believed that the latter emitted in the must seminal seeds, giving rise to the formation of nebulous or new globules, which appeared to have the faculty of reproducing by extension of their own fabric. A similar process took place during the fermentation of sugar. These results led Cagniard de la Tour to conclude that most likely, these grains were organized and belonged to the vegetable kingdom, since they contained nitrogen and were never seen to perform any movements, which could be considered as external signs of will. The globules were extremely small, in the last stage of their development they did not ordinarily exceed a $\frac{1}{100}$ millimeter, indicating that in a cubic millimeter of firm pale yeast there were probably a million or less of these globular individuals. Additional experiments with must led Cagniard de la Tour to conclude that the CO_2 released during fermentation caused the globules to rise to the surface, and that these globules, during their action on their beer wort, decreased in volume, and by this contraction very probably emitted *seminules* or reproductive bodies (Cagniard de la Tour, 1836b).

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Cagniard de la Tour mentioned that Gay-Lussac had written that vinous fermentation still appeared to be one of the most mysterious operations in chemistry, above all because it only took place successively (Gay-Lussac, 1810). Gay-Lussac found that oxygen had a great influence on the development of fermentation in certain liquids, notably grape juice; but that if this oxygen was necessary to develop it, it was not so that it continued. His results indicated that brewer's yeast can produce the fermentation of sugary matters without the influence of oxygen, and that the ferment could be solid in many substances, but in a particular state different from that of brewer's yeast. Cagniard de la Tour kept by Gay-Lussac's procedure, for more than fifteen days above mercury, grape juice that had been squeezed for this purpose from a cluster enclosed under a bell filled with hydrogen gas. Afterwards, he examined part of the deposit left by the juice under a microscope and found it to be almost amorphous. He then introduced a little oxygen under the bell and noticed that it had triggered the vinous fomentation of the grape must and produced many globules. These results led him to assume that the seminules formed part of the material of the deposit, that they did not yet germinate when they were enclosed in the grains of the grape, but that this germination took place as soon as they were exposed to the influence of the oxygen. Cagniard mentioned again that Edwards had found that heating to a suitable temperature egg white diluted with water, resulted in the appearance of globules, which previously did not exist (Edwards and Colin, 1836). He repeated the experiment by putting in a capsule held on a sand bath heated to about 90 °C, a mixture of 50 grams of water and 1 gram of egg white; when part of the albumin had coagulated by heat, he removed the capsule, and after its cooling observed the presence of a surface film, which contained globules of about $1/100$ mm diameter. Another series of experiments were about the fermentation in closed vessels of the juice of currants, grape, and plums, as well as a solution of sugar mixed with egg white. Microscopic examination of the deposits obtained indicated that each one of them consisted in large number of globules like those of brewer's yeast (Cagniard de la Tour, 1838a).

In another experiment, Cagniard de la Tour mixed finely ground dry yeast with solid CO_2 and found that this yeast was equally apt to decompose the sugar afterwards as actively as similar yeast powder, which had not been subjected to cooling (Cagniard de la Tour, 1838a).

Cagniard de la Tours summarized his results as follows: (1) brewer's yeast is a cluster of small globular bodies capable of reproducing themselves, consequently organized, and not a simply organic or chemical substance, as was supposed; (2) these bodies appear to belong to the vegetable kingdom and to regenerate themselves in two different ways; (3) they seem to act on a solution of sugar only as long as they are in a state of life; (4) it is very probably by some effect of their vegetation that they liberate CO_2 from a sugar solution and convert it into a spirit liquor; and (5) they do not perish under the action of considerable cooling or by deprivation of water (Cagniard de la Tour, 1838a).

Pyrolysis of woody materials

Cagniard de la Tour subjected the wood of poplar, sycamore, oak, birch, and boxwood, dried and cut into small cylindrical pieces, to pyrolysis in a sealed glass tube at a temperature of about 350 °C and observed that it first turned brown and then become a very fluid black liquid, which soon thickened as it bubbled up and solidified. In every case, the carbonaceous

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material removed from the tube after cooling, was shiny, broke like glass, and heated to red in the presence of air, burned with a relatively bright flame (Cagniard de la Tour, 1850).

The pyrolysis of wood of guaiac was more difficult to execute and produced a red liquor but took place very easily when carried on with powdered wood mixed with of one-half its weight in water. The resulting carbonaceous was black again. Cagniard de la Tour remarked that although both products had different colors, they behaved like the so-called sticky coal, in the sense that during their combustion they experienced a kind of fusion and produced a flame accompanied by smoke. Grains of wheat did not pyrolyze under the same conditions; they only stuck to each other as they charred. Microscopic examination showed that although deformed, they kept part of their original texture (Cagniard de la Tour, 1850).

Cagniard collected the compressed gas produced during the pyrolysis of birch wood and observed that its volume at room temperature was roughly equivalent to 40 times the capacity of the tube, and that when stirred with water, at least half of it dissolved in it and that the remaining gas was quick to ignite but not very illuminating. The glass tubes used had internal diameter of 2.5 mm and wall thickness 2 to 3 mm; this meant that could withstand an internal pressure of at least one hundred atmospheres.

Additional pyrolytic experiments were conducted with the wood of sycamore trees thirty years old, dried or in the presence of one-half its weight in water. The carbonaceous residue of dry wood produced only a little flame when burning and did not soften, while the one from wet wood was very similar sticky coal, fatty coal and burned with a sooty flame. Cagniard de la Tour assumed that in the second case, part of the wood had probably converted into resin. Other experiments were conducted with fresh sycamore woods five years old. The coals produced were very resinous; under the action of heat, they melted completely and burned with a sooty flame. on burning a flame accompanied by smoke. Cagniard de la Tour remarked that in all cases the lower part the tube was covered with a black bitumen, having the consistency of glue when it came out of the tube; but a few hours after it had been spread out on paper it was dry and formed a rather shiny varnish. Also, the compressed gas produced by fresh sycamore wood exerted only about 15 atmospheres. The lower pressure suggested the formation of ammonia and its absorption by the water. Nevertheless, the remaining water showed a constant acid reaction and sometimes even a rather strong smell of vinegar, which would seem to indicate that the proportion of alkali removed from the tube by this water was not have been significant (Cagniard de la Tour, 1850).

Cagniard de la Tour mentioned that in 1812 James Hall (1761-1832) had heated sawdust and horn in a hermetically sealed rifle barrel and observed the melting of this mixture and its conversion into a kind of artificial coal (Hall, 1812). Cagniard de la Tour reasoned that the high pressure that his glass tubes were able to support, suggested the possibility of using them to repeat Hall’s experiment, without the need of adding horn or other similar flux to lower the temperature, and the advantage of observing the march of the experiment (Cagniard de la Tour, 1851c). For this purpose, he used tubes about 14 cm long, 3 mm internal diameter, and walls 2 to 3 mm thick, and heated them over a charcoal brazier or a bath of boiling mercury. The wood used was previously dried at 100 ^{o}C. The results confirmed his assumptions. He found that in general, very young wood gave a sticky charcoal, and that old wood gave lean coals. Mixing the old woods with water, as described before, produced sticky coals and even a kind of brown resin, partially soluble in ether, and quite like the asphalt used to pave sidewalks (Cagniard de la Tour, 1851c).

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