# WALTHÈRE SPRING AND HIS RIVALRY WITH M. CAREY LEA

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In the spring of 1894, a bitter dispute appeared on the pages of the *Zeitschrift für anorganische Chemie*, concerning priority over investigations on chemical reactions produced by mechanical energy (1-3). Walthère Spring ended the exchange with the following emotional words (3):

> As the circumstances did not allow me to work without interruption, I published my results in several preliminary papers since 1878 … If my plan will be executed by another researcher, like Mr. Carey Lea, his effort will certainly advance science. But whether I must lose all claims to my existing results, I leave for the judgment of my distinguished colleagues. [Translated by the author.]

Who were the opposing scientists? What was the basis of the dispute? Which party was right? What "circumstances" were behind the unusual sensitivity?

Of the two participants, Matthew Carey Lea is usually credited for establishing mechanochemistry as a separate discipline, as he was the first to show that mechanical action can cause chemical changes that are distinctly different from reactions caused by heat. Lea was an independently rich "gentleman scientist," who worked in his private laboratory in Philadelphia for the advancement of science and for his own satisfaction. Of his numerous papers only four dealt with mechanochemistry. They were his last important works, written between 1892 and 1894, when he was about 70 years old. Lea's life and his contribution to mechanochemistry were discussed earlier on the pages of this journal (4) and elsewhere (5).

***Figure 1***. *Walthère-Victor Spring (1848-1911). Downloaded from the Bestor web site (9).*

The accusing party was Walthère-Victor Spring, Professor of Chemistry at the University of Liège, in the French-speaking Wallonia region of Belgium. He was active in several areas of physical chemistry, but his most important topic was the physical and chemical effects of high pressure on various materials and combinations of materials. His first paper on the subject appeared in 1878 (6) followed by comprehensive investigations in the early 1880s (7). Spring did not consider mechanochemistry a fundamentally new discipline as Lea did, but his investigations were more extensive and he started fourteen years before Lea. He continued publishing new results on the subject until 1907 (8), long after the exchange with Lea.

The life and achievements of Spring were described by L. Crismer (10) and a near complete list of his papers was published by E. Bourgeois (11). Only three more recent papers are known about Spring (12-14), but not from the point of view of mechanochemistry. This article focuses on the life of Spring and his research on mechanochemistry. Except for occasional comparisons, the reader is referred to the earlier publications for the details on M. C. Lea's (4, 5).

## The Life of Spring

Walthère-Victor Spring was born in Liège on March 6, 1848 (10); thus he was 25 years younger than Lea. His father, Antoine Spring, was a distinguished professor of medicine at the University of Liège. To his father's dismay, the young Walthère had difficulties at school. He struggled with the Greek and Latin languages and had no interest in medicine. The timid boy felt more comfortable in his workshop, where he became highly skilled in working with wood and metal. Spring could easily have been lost to science without the intervention of his godfather, Jean-Servais Stas, the prominent chemist and good friend of Antoine Spring. He saw promise in young Walthère and became his mentor. With the support and encouragement of Stas, Spring entered the School of Mines of Liège in 1866, from which he graduated with high ranking (10).

From 1871, Spring studied science in Bonn. His chemistry professor was Kekulé, who gave him research projects on polythionic acids and the oxygen-containing acids of chlorine. In physics, he measured the thermal expansion and specific heat of metals and also studied the development of electrical charge on mercury as it flows through capillaries; his advisor there was Clausius. Spring's early results were documented in several publications, beginning in 1873 (11).

The programs in Bonn were excellent, the laboratories well equipped, and frequent new discoveries created an exciting environment. Spring could hardly get better preparation for his later role as professor of chemistry. Compared to him, Lea was an amateur. He learned the basics from a private tutor and studied practical chemistry at Prof. James Curtis Booth's consulting laboratory in Philadelphia, but otherwise he was self-taught (4).

Spring joined the faculty of the University of Liège in 1876 (10). His first assignment was a course in mathematical physics. Although he moved on to more fitting topics after only one year, his ability to teach a subject that far from his areas of interest is evidence of his solid general background. The next year he took over the course of organic chemistry and in 1880 added the chemistry of minerals. He remained responsible for those two fundamental courses in general chemistry for the rest of his life. Spring loved research, but first and foremost he was a teacher, and his first priority was to educate his students to the best of his ability. His courses were designed to the highest standard and he never compromised the quality of his teaching for any reason, not even to free up time for research.

One of Spring's early tasks was to develop teaching laboratories for science students. First he set up a temporary facility, and then in 1880 proposed a new world-class laboratory based on his experience in Germany. His meticulously devised plans were considered overly ambitious by the majority of his colleagues. After bitter, often personal fights, Spring's proposal was rejected and he had to settle for a much smaller and less suitable space with significantly reduced funding. This failure had lasting effect on Spring. He had never been comfortable in society, but now the resentment over this incident and the strained relationship with many of his colleagues made him even more withdrawn. Nevertheless, he played a pivotal role in advancing the chemistry curriculum for science and engineering students. His efforts were often met with hostility: while Spring emphasized the importance of solid scientific foundation, many of his colleagues pushed for more practical, directly applicable knowledge.

He fled from the confrontations at the university to an isolated private life. He was close only to his family and a small circle of friends. (His American rival, Lea, was also living the life of a recluse, but primarily for health-related reasons.) For recreation, Spring enjoyed hiking in the Alps. He marveled the beauty of nature and pondered the forces that created it. When he no longer had the stamina for the long walks, he retreated to his property in Tilft near Liège, where he enjoyed working in his garden (10).

Spring died during the examination period of 1911, on July 17. He needed an emergency tracheotomy, and that led to a pulmonary infection. He was survived by his wife, Jeanne Spring, née Beaujean, and two children, Suzanne and Hermann.

Walthère Spring was elected corresponding member of the Royal Academy of Belgium in 1877 and became regular member in 1887, at age 39. Among his many honors he treasured the honorary membership of the German Chemical Society the most (11).

## Spring's Research

Spring published over 150 papers, of which only about 25 dealt with the chemical effects of high pressure. For a comprehensive review and a near-complete list of publications see Ref. 11.

Spring wrote his first papers in Bonn, under the direction of Kekulé and Clausius. He demonstrated his independent thinking with a paper on the crystallization process, in which he tried to correlate atomic and molecular volume relationships with crystal structure (15). While his ideas were necessarily naïve, the paper demonstrates Spring's desire to explain observable material properties from atomistic principles. His approach of analyzing a broad collection of data to arrive at general conclusions returned in many of his later studies.

Spring was not only fond of nature but also studied it carefully. With the collaboration of Prost, he measured the flow of the river Meuse and the sediment content of its water daily for a full year. From those data he could calculate that about 5 billion cubic meters of water flowed through that river at Liège during the year and it carried a billion and half kilograms of sediment (16). He also studied the climate of Liège and noticed that the temperature in the city was slightly warmer than the temperature in nearby areas. He explained the difference as a local greenhouse effect due to the large industrial carbon dioxide emission (13, 14). This idea was quite original in 1886, when the paper was published. The formation of rocks, not only by pressure, but also by sedimentation and recrystallization from solution, was also a frequent subject of Spring's works (11).

He invested much time and energy into studying the color of water. In the laboratory, he analyzed light that traveled through 26 meters of water-filled tubes. If the water was extremely pure, free from both solutes and floating particles, its color was blue. But the color of water samples from natural sources was always controlled by their impurities. Spring got interested in this subject in 1883 (17) and occasional papers appeared on the color of water and some other liquids until the end of his career (18). While developing methods to eliminate suspended particles from water, he realized that the particles were visible perpendicular to the light ray due to their scattered light, regardless of their very small size. The ultramicroscope developed by Siedentopf and Zsigmondy also used scattered light to detect submicroscopic particles. To Spring's disappointment, they never mentioned his work (11).

## Research on the Effects of High Pressure

Spring's most influential research dealt with the physical and chemical effects of pressure on various materials and combinations of materials. He approached the problem from the point of view of geology, realizing that the high pressure deep inside the earth's crust had to play an important role in the formation of rocks and minerals (10). His interest emerged during his training at the School of Mines and it was reinforced by trips to the Alps. As soon as he got his own laboratory in Liège, he built a compressor and began investigating the compaction and reactions of powdered materials under pressure (6). With varying intensity, he continued the high-pressure studies almost till the end of his life (8). He was always aware of the relevance of his studies to geology. In fact, Crismer rightfully credits him with establishing the "mechanochemistry of geology" (10).

Spring designed and built a compressor using his substantial metalworking experience gained when he was a young boy. The apparatus consisted of a massive lever with a 12.5-fold mechanical advantage, loaded with weights at the far end and pressing on a piston close to the pivot (7). The piston tapered down to only 8 mm in diameter, allowing for pressures up to 25,520 atm, although most experiments were performed below 7,000 atm to avoid permanent deformation of the piston. The compression could be performed in vacuum as indicated by the pumping port shown on Spring's drawing of the apparatus. It is a pity that he did not provide details on the pump and the quality of the vacuum (7).

An unfortunate flaw of Spring's apparatus was that the piston did not fit tightly into the compression cylinder. The gap was a few tenths of a millimeter, sufficient for some material to flow out of the cylinder under pressure. Consequently, his compression was not uniform and uniaxial, but rather an uncontrolled combination of compression and shear. The ambiguous conditions resulted in irreproducible and inconclusive results and a few open disputes (19).

His measurements were interrupted when he accidentally broke his compressor; thus, to secure his priority, he published a short note after compressing only sodium nitrate, potassium nitrate, sawdust and dust from a grinding wheel (6). In order to better mimic the conditions of rock formation, he wetted the powders, expecting that the pressure would remove any excess water. He followed up with a long, comprehensive paper two years later (7). He reviewed ideas on how snow was compacted to ice in glaciers in a lengthy historical introduction, citing observations and explanations by several researchers starting from Faraday. He considered the explanation of Clausius the most plausible: as water expands upon freezing, pressure reduces its melting point. Therefore, compressed snow melts at the asperities (small points of roughness), followed by refreezing as the pressure gets removed by local flow. Water is unique in this respect, as most solids are denser than their melts. Yet, Spring claimed, it could be possible that high enough pressure would increase the interfaces between particles to such an extent, that local atomic movement could result in binding. The process is similar to the flow of a liquid, although it occurs in the solid state. To test this hypothesis, Spring compressed powders of several metals, metalloids, oxides, sulfides, salts and organic materials. The results were mixed, but generally softer materials could be condensed more easily, and Spring attributed this to the larger inter-particle contact surfaces under pressure. He seemed to observe the crystallization of some amorphous materials and the recrystallization of crystalline ones.

The paper described above is mostly about consolidation and not mechanochemical reactions. Yet Spring also tested a few powder mixtures that could react when compressed (7). He expected that pressure would promote or retard the reaction depending on the volume change, according to the principle of Le Châtelier. Indeed, no reaction was observed in a KI + HgS mixture, where the volume would have increased, but a FeS + S mixture reacted readily to form $FeS_2$ with decrease of the total volume (7). In the next two papers Spring reports on studies of the formation of six metal arsenides (20) and eleven sulfides (21) from elemental powder mixtures. Tin reacted with arsenic easily, but the other reactions required several "compressions," meaning that if a powder did not seem fully reacted, Spring repulverized it by filing and compressed the filings again. In some cases, like the reactions of both arsenic and sulfur with silver, up to eight cycles of compressing and filing were necessary to obtain a uniformly reacted block. Unfortunately, it is difficult to identify the roles of the different steps in such a complicated procedure.

The first dispute over Spring's results erupted in 1883, when Jannettaz, Neel and Clermont published a note, claiming that they tried to reproduce some of Spring's results using an apparatus that could produce pressures up to 100,000 atm, but most materials did not crystallize into a solid block (22). Spring was quick to respond. He contacted Prof. Charles Friedel who suggested the investigation and arranged for a demonstration in his laboratory at the Sorbonne. Spring took his heavy compressor to Paris and showed in front of several witnesses including Neel and Clermont, that, if performed correctly, his experiments indeed provided reproducible results. He reported on the successful demonstration immediately (23), and identified impurities and the presence of air as the most probable causes of the falsely negative results. He also pointed out that he never claimed that every powder could be crystallized by pressure. In fact, only 7 of the 83 materials investigated in his study did. Although Spring's rebuttal seemed more than satisfactory, the incident was widely reported (24) and raised lingering doubt over the validity of his results.

Embittered, Spring worked on. He realized that as several compressions were necessary to induce some metal-sulfur reactions, it was natural to ask exactly how much sulfide formed during each pressing-refiling cycle. He performed chemical analysis after each compression on mixtures of Ag, Pb and Cu with sulfur (25). In each case, the reaction took place gradually; only a few percent reacted during the first pressing and the yield was less than 70% even after six. He mentioned that the incompleteness of the reaction agreed with the observation of Jannettaz, maybe to mend fences with his colleagues in Paris. According to Spring's assessment, pressure was not a chemical agent, but a facilitator that increased the interfaces between the powder particles and thereby intensified chemical interaction.

Spring extended his studies from simple combination reactions to the exchange reaction between barium sulfate and sodium carbonate; the resulting papers are his most cited works (26, 27). In order to quantify the observed changes, he performed chemical analysis that required separating the water soluble and insoluble components by washing. Unfortunately, the presence of water affected the reaction much more than compression did; thus his analysis reflected the state after water was added and not the composition of the dry powder. Correction for the effect of water could not eliminate this problem (19).

A new dispute erupted in 1887, now with William Hallock of the U.S. Geological Survey. In a short and pointed paper, Hallock showed that solids do not liquefy under pressure, but flow in the solid state under large enough load (28). Spring was quick to point out that he never meant true melting, but flow in the solid state that resembled the flow of a liquid. His words were misunderstood and misrepresented (29). This could be true, but misunderstanding is often the consequence of unclear language and Spring's papers often lack clarity. This is understandable in a new research area where the terminology is still ambiguous, but it did result in problems. Hallock was ready to retract to avoid further conflict (30). But he also pointed out that many effects attributed to pressure by Spring were more likely the consequence of kneading due to the uneven distribution of pressure in his compressor or of regrinding the product. Spring's reply is probably the clearest and most compact formulation of his fundamental beliefs: "…pressure is not a chemical agent to the same extent as heat or electricity." It promotes the reaction between particles by increasing the contact surface and kneading is just another way of bringing surfaces into intimate contact, but the reaction itself takes place by ordinary diffusion. The time dependence of some reactions also suggested that diffusion was at play (31).

## The Priority Dispute with M. Carey Lea

Spring's interest turned to other subjects during the late 1880s and early 1890s, but when he read Lea's paper on reactions induced by grinding that completely ignored his work (32), he decided to raise the question of priority. Interestingly, the dispute between the American Lea and Belgian Spring played out in the German *Zeitschrift für anorganische Chemie*. This is not an accident: Spring published primarily in Belgian and French journals, but he also wrote summaries and sometimes independent papers in German. Sometimes, like in the case of Ref. 21, the French and German versions differ so little that they can hardly be considered separate publications. Spring was also a member of the editorial board of *Zeitschrift für anorganische Chemie* (11). Lea's approach was quite different. He published his important papers simultaneously in the *American Journal of Science*, in *Philosophical Magazine* (identical except for the British spelling) and in German translation in *Zeitschrift für anorganische Chemie*. Between Spring and Lea, German was the common language. Interestingly, there is no record of the dispute in any other journal.

Lea had given Spring general credit for his work in mechanochemistry in his previous paper (33). But the article that raised Spring's ire (32) was strictly about endothermic reactions, and Spring never even mentioned that the exothermic or endothermic nature of a reaction could make any difference in how a system responds to mechanical agitation. Also, the note claiming priority over Lea (1) contradicts itself, in that he restates that the primary process in mechanochemical reactions is diffusion at the interfaces between particles and the reactions proceed toward chemical equilibrium as usual, while the essence of Lea's claim was that the continued supply of mechanical energy was required to bring about endothermic reactions. (Whether Lea's idea about energy transfer in exothermic and endothermic reactions is correct is another matter.) Spring also remarked that he would continue his long-term plan of experiments "as soon as conditions would permit." Maybe he was overwhelmed by his teaching duties, although he was publishing regularly on other subjects.

Lea was surprised by Spring's claim and refuted it by stating that his objectives, experiments and conclusions were entirely different from those of Spring (2). He reiterated that the possibility of inducing exothermic reactions by mechanical energy has been known for quite some time, but doing the same for endothermic reactions was widely considered impossible. In that sense, he considered his results fundamentally new, while the results of Spring were just further examples of a well-known fact.

Spring's reply immediately followed Lea's note (3). There he said that his "claims were not specifically about one or the other fact of the question but about the topic itself." He considered himself the first to carry out broad systematic investigations on the effects of mechanical action, specifically high pressure, on materials. On that account he was right. He also repeated his complaint about his "circumstances." He said the he was not able to work without interruption, but wanted to assert his ownership of at least the already published results.

Both Lea and Spring continued working after this incident. Lea published only one more paper on mechanochemistry, then moved on to other subjects and died three years later. Mechanochemistry never regained the central position in Spring's research to the degree it enjoyed in the early 1880s. But he did publish a few more papers on the subject, one right after the exchange, probably to demonstrate his continued interest in mechanochemistry (34). His last paper on chemical changes caused by mechanical deformation was published in 1907 (8), only four years before his death.

## Spring's Legacy in Mechanochemistry

What is the place of Walthère Spring in the history of mechanochemistry? He was unquestionably the first person to carry out wide-ranging experiments on the compaction of powdered materials under pressure, with a close eye on their implications for geology. He also studied combination reactions and decomposition due to pressure. His questions were revolutionary and the breadth of his studies unparalleled in the 1880s.

The validity of his conclusions is a different matter. Johnston and Adams reviewed the literature on the effects of pressure on the physics and chemistry of solids (19). They were aware of the controversies and opposing views in the area and intended to be "as impartial as may be." Necessarily, they paid substantial attention to the works of Spring. One after the other, they showed that his methods were flawed and his conclusions incorrect. Many, although not all, problems were caused by Spring's leaky cylinder, that never produced uniform compression. This paper appeared in 1913, two years after Spring's passing. Whether the authors delayed the publication intentionally, or it just happened this way, is impossible to tell. Either way, it certainly avoided another exchange of harsh words.

Some of Lea's results are still frequently cited as clear proofs that the chemical effects of mechanical action are different from the effects of heating (4). His results were not only unique at their time, but they are still considered technically correct and one of the clearest demonstrations of the difference between mechanochemical and thermochemical reactions. Accordingly, he is rightfully considered the "father of mechanochemistry." On the other hand, Spring's results were disproved and the current ideas about the effects of pressure are essentially different from his way of thinking. But his works inspired substantial activity, thus they contributed positively to the development of mechanochemistry, especially from a geological point of view. Therefore, he also deserves his place among the early practitioners of mechanochemistry.

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### 2019 to be International Year of the Periodic Table

In December 2017, the 72nd session of the United Nations General Assembly proclaimed 2019 to be the International Year of the Periodic Table. 2019 marks the 150th anniversary of Dmitri Mendeleev's first version of the periodic table. Observations are being planned by UNESCO, IUPAC (which is also celebrating its 100th anniversary in 2019), and ACS among other organizations.