KLAUS AT KAZAN: THE DISCOVERY OF RUTHENIUM (1)

David E. Lewis Department of Chemistry, University of Wisconsin-Eau Claire, Eau Claire, WI 54702-4004, USA; lewisd@uwec.edu

Ruthenium was once called the "little Benjamin" of the platinum group metals (2), referring to its lesser status compared to the other platinum metals. For much of the twentieth century there were, indeed, remarkably few papers dealing with this element and its compounds (3). Then things began to change, and we can illustrate this by noting that three Nobel laureates—Henry Taube, who studied electron transfer reactions in metal complexes, Robert Grubbs, whose work helped make olefin metathesis a major synthetic method, and Ryoji Noyori, who developed practical chiral catalytic hydrogenation catalysts—all used ruthenium as part of their prize-winning work. By the time that this paper is being written, ruthenium has, in many ways, become a modern "miracle" metal. Certainly, the number of papers published following the first half of the twentieth century dramatically increased—the number of papers appearing during the decade of the 1980s approximately doubled the output of the previous decade, and each subsequent decade has seen an approximately 50% increase in the number of published papers devoted to aspects of the chemistry of this remarkable element (3). While part of the increase may be attributed to the general proliferation of journals now available, this by no means accounts for more than a minority of the increase.

Ruthenium metal itself was first isolated pure (4) in 1844 by Karl Karlovich Klaus (Клаус Карл Карлович, Carl Ernst Claus, 1796-1864, Figure 1) (5), who was, at the time of its isolation, Extraordinary Professor of Chemistry at Kazan University. The university itself had been established forty years earlier by royal decree of Tsar Alexander 1, and was at the time the easternmost university in Russia. At the time of his discovery, Klaus was relatively unknown—certainly, he did not have the international reputation as a chemist that the discovery of ruthenium gave him, nor had the Kazan University School of Chemistry achieved the stature it would attain over the next half century as an important center for chemical research and education.

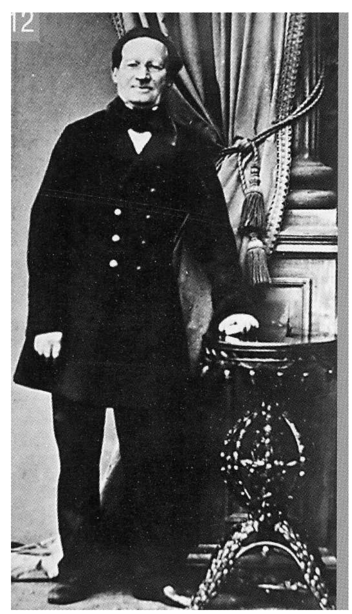

***Figure 1**. Karl Karlovich Klaus (Клаус Карл Карлович, Carl Ernst Claus) in Dorpat (daguerreotype, ca. 1852).*

Klaus was born in Dorpat (now Tartu, in Estonia) to Carl Claus, a talented painter of Baltic German descent who christened his son Carl Ernst (Karl Karlovich; in Russia, the patronymic, "Karlovich" (son of Karl) displaces the middle name in German). It is perhaps fortunate that the son inherited some of his father's talent as an artist, because the arts allowed him a place to escape from the trauma of his childhood. His skill as an artist is evident from some of the illustrations in this paper, which are his work. Klaus' childhood was, indeed, traumatic. At age four, he lost his father, and his mother married again, becoming the wife of another artist; a scant two years later, Klaus' mother also died, and he was left an orphan—unloved and neglected—in the home of his "hated stepfather." Klaus received little support or love in this household, and this neglect of a lonely child may have been important in building his self-reliant and persistent character.

***Figure 2**. "Saratow" by K. K. Klaus*

Although Klaus was enrolled in the Gymnasium at a young age, he did not have (or his stepfather did not give him) sufficient funds to enable to complete the course of study, and he had to begin supporting himself by the age of 14 years. Thus, in 1811 he left Dorpat for St. Petersburg, where he became the student of an apothecary.

Klaus was a voracious reader, and during his time as a student with the apothecary he was able to teach himself pharmacy, botany, and chemistry from the books that he read. So successful was Klaus at educating himself, that—despite his lack of a formal education—he was able to pass the examinations for Assistant Pharmacist, administered by the St. Petersburg Medical-Surgical Academy in 1815. As a result, he became the youngest qualified Assistant Pharmacist in Russia (6). In 1816, while still working at the pharmacy in St. Petersburg, he took the test at Dorpat University to qualify as Provisor in pharmacy (later, this title changed to kandidat in pharmacy (7)), which he passed. The next year, the Medical-Surgical Academy also conferred on him the title of Provisor, and he moved to Saratov (Figure 2) in that capacity, as a fully qualified pharmacist.

Klaus spent the next ten years in Saratov. During his time there, he married Ernestine Bate, whom he had known since childhood. The marriage was a happy one, and the couple eventually had four children: three daughters, born at Kazan, and a son born in Dorpat after Klaus had left Kazan. At Saratov, Klaus was successful enough to save the money required to move to Kazan (Figure 3) and establish his own pharmacy there. He did so in 1826.

***Figure 3**. A view of the city of Kazan from the direction of Kaban Lake, 1828, by K. K. Klaus*

His attention to detail, his breadth of knowledge in general, and his wide knowledge of medicinal herbs in particular, quickly made his pharmacy the most respected in Kazan. This had the result of allowing Klaus to enter the intellectual circles of the city. At the same time, he also obtained an excellent reputation as a scientist, based on his studies of the Volga-Ural flora. In fact, there are several species of plants that bear his name (8).

While he was a pharmacist in Kazan, Klaus was approached by the Professor of Zoology, Eduard Aleksandrovich Eversman (Еверсман Едуард Александрович, Eduard Friedrich Eversman, 1794-1860, Figure 4) to accompany his expedition to study the flora and fauna of the Volga region. Given Klaus' interests in pharmacy and the use of natural herb medications at the time, it is not surprising that he accepted the invitation.

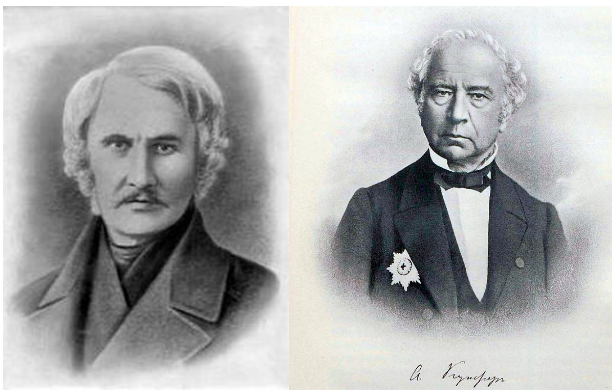

Figure 4*. Eduard Friedrich Eversman (Еверсман Едуард Александрович, left) and Adolf Yakovlevich [Adolph Theodor] Kupfer (Купфер Адольф Яковлевич, right).*

The journey covered the area from Kazan to a number of places in the Orenburg district of Astrakhan province, as well as the shores of the Caspian Sea. This expedition, which took place during 1827, eventually led to Klaus' publication of the work in the German language (9). A year later, Klaus accompanied Adolf Yakovlevich Kupfer (Купфер Адольф Яковлевич, Adolph Theodor Kupfer, 1799-1865, Figure 4), the Professor of Chemistry and Physics at Kazan University, on his expedition to examine the placer deposits of platinum and gold in the Ural region (10). Kupfer asked Klaus to accompany him, and wrote later, "I was accompanied by Klaus, the Kazan pharmacist, an outstanding artist, whose paintings graced the appearance of that work" (11).

This expedition with Kupfer was a watershed moment in Klaus' life, because it spurred his interest in the chemistry of the platinum metals, and an eventual desire to complete his formal education in chemistry by returning to Dorpat to obtain his degree. Three years later, Klaus sold his pharmacy for what he felt was half its true worth, and he returned to Dorpat to obtain his degree in chemistry. From 1831-1837, he was an Assistant in Chemistry at Dorpat, serving for a time as the assistant to another Dorpat native, Gottfried Wilhelm Osann (Озанн Готтфрид Вильгельм, 1797-1866, Figure 5). At the time, Osann was Professor of Chemistry, and a recognized expert in the chemistry of the platinum metals. He was working on the residues from the platinum ores that he had obtained from the Finance Minister, Count Yegor Frantzevich Kankrin (Канкрин Егор Францевич, 1775-1845) (Figure 5), and it is almost certain that Klaus learned the techniques for analyzing platinum ores from Osann.

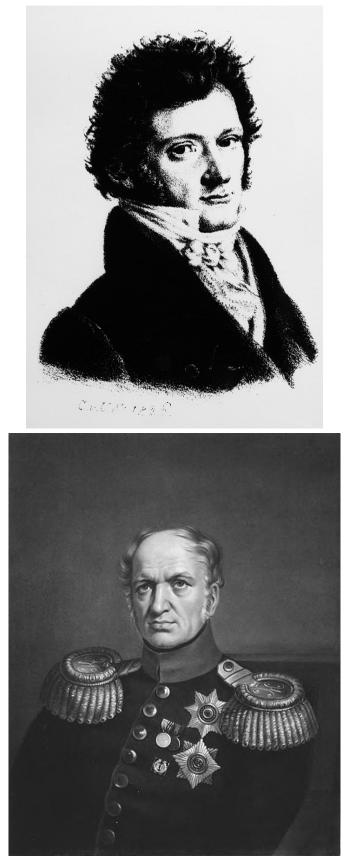

Figure 5*. Gottfried Wilhelm Osann (Озанн Готтфрид Вильгельм, top) and Georg Ludwig, Graf von Cancrin (Граф Канкрин Егор Францевич, 1774-1845, bottom).*

At this time, Russia used platinum (which was known as "white gold") as a coinage metal, and Count Kankrin, who spearheaded the reforms of the Russian financial system during his twenty-year tenure as Finance Minister, was concerned that there were significant amounts of platinum still left in the residues from the process of refining. Consequently, he sought to determine how much platinum remained in the ore residues, and if methods for its extraction could be developed. As part of his efforts to determine the amount of platinum in the ores from the Urals, he sent samples of the ore to institutions within the Russian empire and abroad. Among those who received samples was the great Swedish chemist, Jöns Jacob Berzelius (1779-1848). Four pounds of the platinum ore were also sent to Dorpat, where they were analyzed by Osann.

Osann subjected the crude platinum to the standard methods of analysis of the day: the crude mass was dissolved in aqua regia, and the insoluble material was removed by filtration. The filtrate was then treated with ammonium chloride to precipitate the ammonium hexachloroplatinate (IV), along with the corresponding iridium (IV) complex. On heating, this salt decomposed to a platinum-iridium sponge that could be compressed into a malleable metal for minting coins (12). This crude platinum metal could be further treated by redissolving it in aqua regia; in this case, the iridium remains as an insoluble black solid.

In the course of his researches, Osann obtained what he considered to be three new elements, which he named ruthenium (L. *ruthenia*: Russia), pluranium (combining the initial letters of platinum and Urals), and polinium (Gk. polioς, grey); Osann later suggested that his polinium may, in fact, be impure iridium (13). He sent samples of his new elements to Berzelius, but the great chemist and mineralogist failed to confirm his discovery. Perhaps as a consequence of this, Osann withdrew his claims a year later (14). A later (1900) assessment of Osann's work (15) concluded that his polinium was impure iridium, perhaps containing some ruthenium, and that the red needles of ruthenium in his first report (13) may have been a mixture of osmium and ruthenium tetroxides; in 1845, Osann agreed with Berzelius that the crystals with a golden luster, to which he had transferred the identity as ruthenium, were probably a mixture of zirconium, iron, silicon and titanium oxides, but he vigorously defended the identity of his polinium with Klaus' ruthenium (16). The identity of pluranium has never been satisfactorily established. In a recent biographical account (17) containing a re-evaluation of his claims, Hödrejärv asserts that Osann had, in fact, obtained ruthenium as he had claimed, but he concedes that Osann's claims were undermined by the irreproducibility of his results. A succinct account of the "discovery" of the elements polinium and pluranium has recently been published (18).

While a student at Dorpat, Klaus accompanied Professor Carl Christoph Traugott Friedemann Goebel (Гёбель Карл Христиан Траугот Фридеман, 1794-1851, Figure 6) in his expedition to the Volga steppes, in part because Klaus spoke and read Russian. Goebel wrote a report of this expedition (19) in two volumes on his return to Dorpat; the work received a lesser Demidov Prize (the smaller version of the most prestigious award conferred by the Academy of Sciences), although Klaus' share of this prize was never confirmed. In 1835, Klaus passed the examinations for the kandidat of philosophy degree at Dorpat University, and two years later, he submitted his dissertation (20), and passed the examinations—which included questions about the chemistry of the platinum metals—for the degree of Master of Philosophy. In May the same year, Klaus read the required sample lecture ("On a Rapid Method for Preparing Chemical and Pharmaceutical Products") at the St. Petersburg Medical-Surgical Academy, and in June, 1837, he was offered a position as Adjunct in Pharmacy at Kazan University.

Figure 6. Carl Christoph Traugott Friedemann Goebel (Гёбель Карл Христиан Траугот Фридеман, left) and Nikolai Nikolaevich Zinin (Зинин Николай Николаевич, right).

Klaus took up his pharmacy appointment at Kazan in August 1837, during the time that the Adjunct in Chemistry, Nikolai Nikolaevich Zinin (Зинин Николай Николаевич, 1812-1880, Figure 6), was abroad on his *komandirovka*. At the same time, the chemistry laboratory was being moved into a new building, which was expanded in phases from 1837 to 1839 thanks to the efforts of the Trustee of the Kazan educational district, Count Mikhail Nikolaevich Musin-Pushkin (Граф Мусин-Пушкин Михаил Николаевич, 1795-1862, Figure 7). Musin-Pushkin had himself been a student at the university, and strongly supported it. One of his most significant acts was to secure the appointment of the mathematician, Nikolai Ivanovich Lobachevskii

(Лобачевский Николай Иванович, 1792-1856, Figure 7), as Rector of the university.

***Figure** 7. Count Mikhail Nikolaevich Musin-Pushkin (Граф Мусин-Пушкин Михаил Николаевич, top) and Nikolai Ivanovich Lobachevskii (Лобачевский Николай Иванович, bottom).*

The expansion of the chemistry building required someone to oversee the routine day-to-day work supervising the transfer to the new building. But the Adjunct, Zinin, was not available to do this, so Klaus was seconded to the *kafedra* of chemistry at the request of Professor Ivan Ivanovich Dunaev (Дунаев Иван Иванович, 1788-1840). This gave him the responsibility for fitting out and maintaining the chemistry laboratory, and for conducting experiments in inorganic and pharmaceutical chemistry. In order to obtain the rank of Extraordinary Professor of Chemistry, Klaus required the degree of *Dr. Filosofii*, which he obtained in 1839, with a dissertation (21) describing a study of the compounds in the mineral waters of the Sergievskii district. On graduating with this degree, he was appointed Extraordinary Professor of Chemistry.

In 1840, after receiving his appointment in chemistry, Klaus received an allotment of platinum residues from Count Kankrin, and began his analysis using the protocols that had been established by earlier workers in the field (especially Osann). He spent the next two years in completing the analysis of these residues for the known platinum metals, and was able to extract more platinum from these residues. His work had been fruitful enough for Count Kankrin to award him a further allotment of 8 kg of the residues, as well as 300 g of platinum metal. Unfortunately, this second batch of the spent ore contained much less platinum, and he was forced to inform Kankrin that the second batch of residues had so little platinum that it was of scientific interest only (22).

It is probably well here to dwell for a brief time on Klaus' laboratory practices. Some of his habits in the laboratory would certainly attract the serious attention of any modern chemical safety officer because of their likelihood of harming his health. Among other things, he tasted practically all his solutions: his colleagues recalled that on arriving in the laboratory early in the morning, Klaus often tasted solutions of substances with which he had to work that day. Thus, after dissolving platinum ore in aqua regia, he determined the strength of unreacted acid by dipping a finger directly to the reaction mixture, and then touching it to his tongue. When he first isolated osmium tetroxide, he noted that it had a sharp and peppery taste, and reminiscing later about working with osmium compounds, he wrote, "... osmic acid belongs among the most harmful substances ... I suffered much from it ..." (23).

This assessment came at a painful price. At least twice, Klaus had generated enough osmium tetroxide to flood the laboratory with its vapors and cause serious injury: In April 1844, a release of the gas into the laboratory, generated while he was alloying 15 pounds of platinum residues with nitre, injured some retired soldiers who were not even inside the laboratory; it took them three days to recover, even though they were outside in the hall. The *Preparator*, Gelman, was inside the laboratory, and was therefore exposed longer; he suffered a dangerous inflammation of the lungs. Klaus escaped serious injury on this occasion by being able to exit from the laboratory quickly into the auditorium (24). A year later, he did not escape injury as he had in 1844, and the poison affected him so much that he was forced to stop his experiments for two weeks (25). As he was characterizing his new element, he tasted an ammonium complex of ruthenium. This left him with a mouth so badly blistered that he was forced to take three weeks off from his research and teaching (5b). None of these incidents, however, was able to deter him from the practice of tasting his solutions; instead he impatiently endured these forced hiatuses, only to return to the research laboratory as soon as he had recovered.

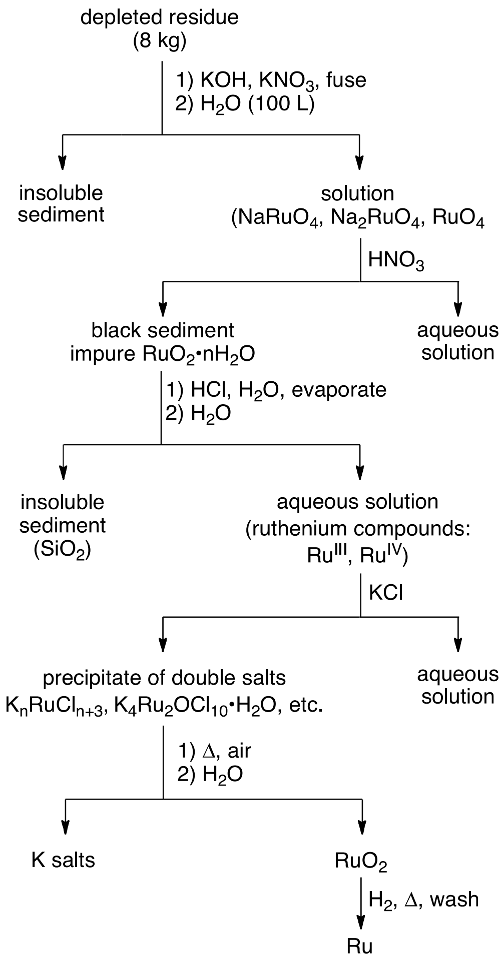

Figure 8*. Klaus' procedure for the isolation of metallic ruthenium from the poor platinum residues of the Ural placer platinum ores.*

Klaus now turned his attention to the metallic residue that was insoluble in aqua regia, and by means of the sequence of steps in Figure 8, he was able to obtain pure ruthenium metal for the first time. In Figure 8, the ruthenium compounds are identified as Klaus designated them. His procedure led him to believe that among the double salts precipitated with potassium chloride, he had isolated a hexachlororuthenate, K_2RuCl_6 analogous to the corresponding hexachloroplatinate salt, K_2PtCl_6. Fifty years later, Howe showed that this salt was, in fact, the nitrosopentachloro complex, K_2RuCl_5NO (26).

Klaus' habit of ignoring potential hazards to his health provides the clue to how he was able to track this new metal. As an inveterate taster of his solutions, he simply followed the "strange" taste in his metal solutions, and the associated acrid odor of ruthenium tetroxide. His senses of smell and taste were probably the most sensitive analytical tools available to him, even though the most hazardous to use.

Klaus first reported the isolation of his new metal in Russian in the *Uchenye Zapiskii Kazanskogo Universiteta* [*Scientific Notes of Kazan University*], taking one complete issue of the journal in 1844 to describe his results (4). The same year, he reported the discovery of ruthenium—now in German—in the *Bulletin de la Classe Physico-Mathématique de l'Académie Impériale des Sciences de St. Petersbourg* (27), and he was promoted to Ordinary Professor of Chemistry at Kazan. In 1845, Klaus reported his discovery in the *Gorny Zhurnal* [*Mining Journal*], one of the oldest journals in Russia. In this paper, he wrote (28), "At the very beginning of the work I noticed the presence of a new substance, but at first I could not find a way to separate it from impurities ... This new metal, which I have named ruthenium in honor of our Fatherland, certainly belongs among the most interesting materials." It is worth noting that Klaus considered Russia, and not Germany as his homeland, although he used the German term, "Fatherland," instead of "Motherland," or "Mother Russia."

Klaus is reported to have been an excellent lecturer, and to have spoken Russian with a "fair" accent. However, he is also reported to have lapsed into the German of his boyhood whenever he became excited or inspired (29). His choice of words here may reflect that although Dorpat was under the rule of the Tsar, it retained its German language and traditions until the late nineteenth century. As part of the University Statute of 1884, Alexander III promoted the Russification of all universities in the empire and repealed many of the reforms of his predecessor. This included establishing Russian as the official language of education (30).

The same year, reprints of his discovery appeared in a number of western European journals. The version of his paper in the *Bulletin* that was published in *Poggendorff's Annalen der Physik* drew an immediate response from Osann, who claimed that Klaus' ruthenium was actually his polinium (16); this, in turn, received an immediate response from Klaus defending his priority for the discovery of the metal. As part of this effort, he published a comparison of the properties of the new metal and iridium (31). In addition to publishing the work in

Russian, Klaus work was communicated to Paris by Germain Henri Hess (Гесс Герман Иванович, 1802-1850) (32). This paper, likewise, was reprinted in German and abstracted in English.

After obtaining his sample of ruthenium as a grey powder, Klaus sent samples of the new metal and some of its salts to Berzelius for authentication of his discovery. Predictably, perhaps (given his history with Osann's three "elements" from the same source), Berzelius was skeptical, and initially dismissed the claims. However, Klaus was dogged in his insistence that he had discovered a new metallic element. A year later, Berzelius wrote him a letter where he described the new element as being an impure form of iridium, and Klaus immediately replied that—respectfully—he could not agree with that assessment (33).

It appears that his letter crossed Berzelius' second letter in the post because just eight days after sending the letter with his initial negative assessment, Berzelius again wrote to Klaus, this time confirming his claims of the discovery of a new element, and praising the way in which Klaus had acknowledged the earlier work of Osann (34). This letter ended the doubts of at least some of the European skeptics of Klaus' discovery. Berzelius concluded this letter with the following: "I have taken the liberty of submitting an abstract of your article from the Academy of Sciences to the editor, who will print it in a report of this meeting." The report appeared in the *Jahresberichte* in 1846 (35).

This same year, Klaus received the Demidov Prize for his work in the chemistry of the platinum metals and the discovery of ruthenium. In 1847, by which time his priority as the discoverer of ruthenium had become generally accepted, Klaus published a summary of his contributions to the chemistry of the platinum metals, including the discovery of ruthenium (36).

In 1852, Klaus was offered the Chair of Pharmacy at Dorpat University. The reasons for this move were personal, and based mainly on family and financial considerations. His second daughter and her husband and son lived in Dorpat, and Klaus bemoaned the fact that his salary could not support his family both in Kazan and at Dorpat. The solution to the problem was to unify the family in one city, which set the two sides of Klaus' character in conflict with each other. Klaus, the patriarch of the family, was needed in Dorpat, while Klaus, the scientist, had strong ties to Kazan. As we know, his family concerns won, and he moved to Dorpat. One other thing that may have facilitated this move was the opportunity to occupy the Chair of Chemistry, which had been vacated by the death of Göbel. Klaus had written to Alexander Georg von Bunge (Бунге Александр Андреевич, 1803-1890), who had been at Kazan, and who taught botany at Dorpat, asking him to help facilitate his application. Although he did not receive the Chair in Chemistry he sought at Dorpat, he was appointed to the new Chair of Pharmacy.

Klaus left Kazan in 1852, but he did not completely break his ties there. In 1854 he wrote an account of the chemistry of the platinum metals as a *Festschrift* for the fiftieth anniversary of the founding of the University (37). This monograph was to define the field for the next century (5g). It had been Klaus' intention to complete a full monograph on the chemistry of the platinum metals, but he never accomplished this during his lifetime, and sections of his proposed manuscript were never completed. However, the parts of the manuscript that he did complete, were gathered by his student, Butlerov, and published after his death (38); the manuscript had languished among his papers for a decade, and parts of the manuscript had been lost during the decade between its writing and its publication.

Following his move to Dorpat, Klaus continued with his work on the platinum metals, and he continued to publish in the area. He traveled extensively, and received numerous honors throughout Europe; Pitchkov (5g) has provided an excellent synopsis of these travels and honors. In 1861, Klaus was elected as a Corresponding Member of the Russian Academy of Science. During his return from St. Petersburg to Dorpat after he had attended a meeting of the St. Petersburg Pharmaceutical Society as an honored guest, Claus caught a chill that failed to improve; how much this may have been due to the damage to his lungs from osmium and ruthenium tetroxides must remain conjecture, but it seems quite reasonable to propose that this may well have predisposed him to pneumonia. He died shortly after his return to Dorpat.

Acknowledgments

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I. S. Antipin for their support. I am also indebted to a reviewer for help with the series of references between 1844 and 1847, when Klaus announced the new element.

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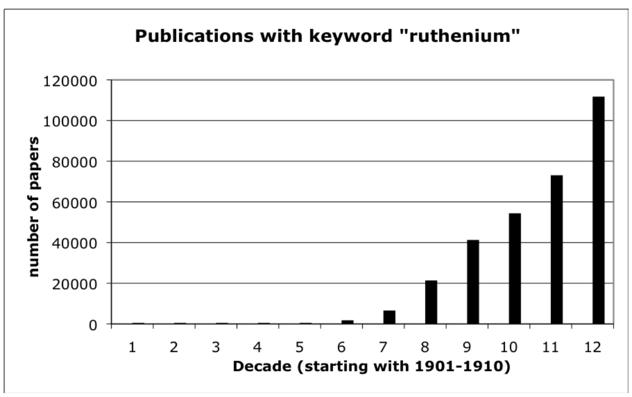

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About the Author

David E. Lewis is a frequent contributor to the *Bulletin*. He holds Ph.D. and D.Sc. degrees from the University of Adelaide, and is a Fellow of the Royal Australian Chemical Institute and the Royal Society of Chemistry. His works have recently appeared in a bilingual edition in Russian and English: Каанская химическая школа глазами американсчого историк химии. Сборник работ Джвида Э. Льюиса. *The Kazan School of Chemistry Through the Eyes of an American Historian of Chemistry. Collected Papers by David E. Lewis* (Kazan University Publishing House: Kazan, 2016). When not researching the history of organic chemistry in Russia, he is a synthetic organic chemist and the author of a new textbook: *Advanced Organic Chemistry* (Oxford University Press: New York, 2016).