Rediscovery of the Elements

Columbium and Tantalum

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Charles Hatchett (1765–1847), a prosperous London coach-builder and avocational chemist, discovered columbium (niobium) in 1801. The following year, Anders Gufstaf Ekeberg (1767–1813), professor of chemistry at the University of Uppsala, Sweden, discovered tantalum. Because of the chemical similarity of the two metals (both 5B members of the Periodic Table), they were believed to be the same element for half a century (Figure 1).

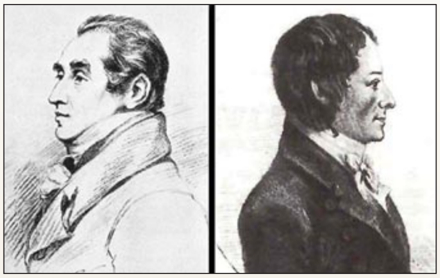


Figure 1. Charles Hatchett (left) discovered columbium (today known as niobium) in 1801 (engraving by F. C. Lewis after Thomas Phillips' painting, now lost), and Anders Ekeberg (right) discovered tantalum the following year (engraving by A. U. Berndes, after a drawing by J. E. Roos). Wollaston's work in 1809 suggested the two elements were identical, and Hatchett and Ekeberg died never knowing they had in fact discovered different elements.

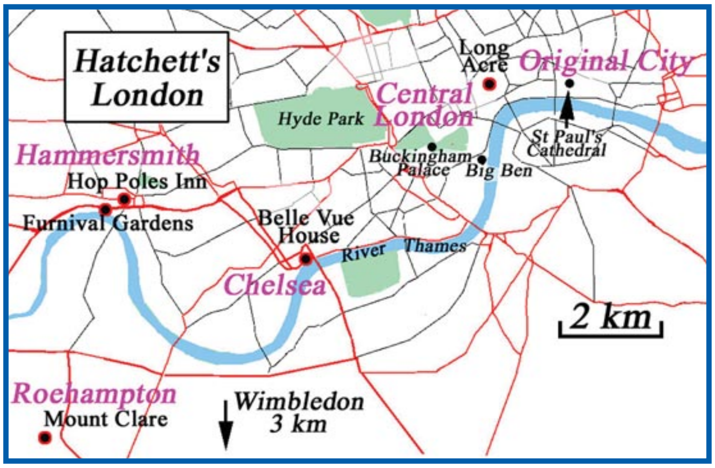


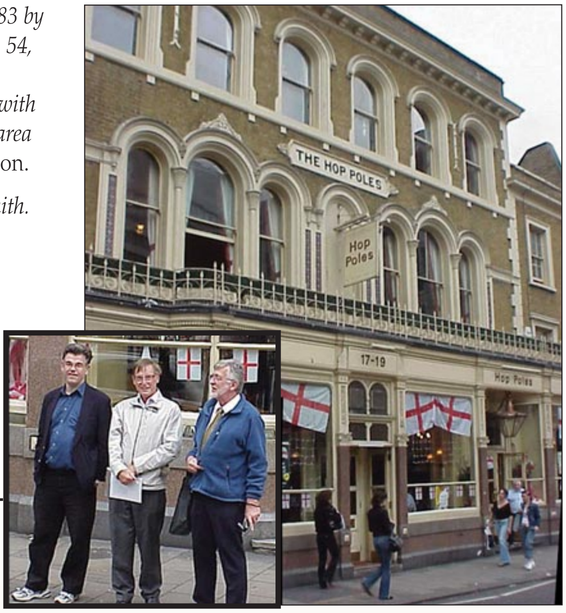
Figure 2. Hatchett's coach-making business was located at 121 Long Acre (N51° 30.76 W00° 07.52). Charles spent his youth in Belle Vue in Chelsea (owned by his father) and then repurchased the home for his final years (91-92 Cheyne Walk; N51° 28.92 W00° 10.45). Charles Hatchett's first home after his marriage was at the Lower Mall in Hammersmith at an address today unknown; this area was bombed in World War II and is now occupied by Furnival Gardens (N51° 29.42 W00° 13.99); this is where he discovered columbium. Also in Hammersmith may be found today at Hop Poles Inn (17-19 King Street, N51° 29.56 W00° 13.55), a favorite haunt of Charles Hatchett. In mid-life his home was Mount Clare (1808–1819) at Minstead Gardens, Roehampton (N51° 27.11 W00° 15.04).

Charles Hatchett[1] (Figure 2) was born at his father's carriage manufactory on Long Acre (Figure 3). Charles, being the only son, had financial resources for pursuing many hobbies, including music, art, travel, and the sciences. Charles married Elizabeth Martha Collick (1756–1837), daughter of a wigmaker. They traveled for two years in Poland and Russia and then returned to Hammersmith, where his father had extensive property (Figure 4). In 1790 Charles was sent to St. Petersburg to deliver a coach to Catherine the Great. During this trip, he made extensive visits to scientists, mines, and mineral collections. One of these scientists was Martin Klaproth in Berlin, the discoverer of uranium.[2g] Absorbed in his new passion of chemistry and mineralogy, Hatchett continued his scientific tours through Europe and by 1796 published his first paper, a significant analysis of lead molybdate[3a] (distinguishing it from lead tungstate). He became a member of the Royal Society, the Royal Institution (to which he frequently made donations), the Linnean Society, and the Mineralogical Society.

In the world of chemistry Hatchett seemed to have a sense for knowing what might be important to investigate. His first paper on lead molybdate[3a] was deemed important enough to merit his election to the Royal Society. His work with bones and shells[3b] first determined the different compositions of bones and teeth (mostly calcium phosphate) and shells (mostly calcium carbonate).[3b] But his most important discovery was columbium (niobium).

The discovery of columbium. The discovery of this element was prompted by Hatchett's curiosity regarding a certain ore in the British Museum. As he was arranging specimens in this museum's mineral collection, he noted a "heavy black stone" originating from "Mr. Winthrop, of Massachusetts."[3c] Hatchett chose well—this was a relatively pure sample of columbite, with the (ideal) composition $(Fe,Mn)Nb_2O_6$, ready for a straightforward chemical analysis.

Hatchett, in his Hammersmith home/laboratory (Figure 5), took 200 grains (13.0 grams) of the sample and reacted with "carbonate of potash" at "red heat." The resulting grayish-brown mass was taken up in boiling distilled water and treated with nitric acid, whereupon a "copious, white flocculent precipitate" was thrown down. The filtrate was proven to be "chiefly of iron" by (1) its reaction with "prussiate of potassium" (potassium ferrocyanide) to



TOP LEFT: Figure 3. This is the appearance of Hatchett's coach-making business (as drawn in 1783 by John Miller) when Charles Hatchett was 18, and when his father John Hatchett (1729–1806) was 54, at the prime of his career with a company that employed several hundred workers. Charles was apprenticed here when he was 14. By 1791 there were more than 25 coach-makers on Long Acre, with several dozen supporting trades (leatherworking, woodworking, etc.). In the twentieth century the area shifted to motor carriage manufacture. Courtesy, London Metropolitan Archives, City of London.

RIGHT: Figure 4. Hop Poles Inn, which Charles Hatchett frequented when he lived in Hammersmith. John Hatchett had his mansion nearby when he was a magistrate of Hammersmith.

INSET: In front of the Hop Poles Inn are (left to right) Peter Morris, William Griffith, and Jim Marshall. Drs. Peter Morris (Senior Curator, Chemistry, Science Museum at South Kensington) and Bill Griffith (Chemistry, Imperial College) are authors of the excellent biography of Charles Hatchett[1] extensively referenced herein.

TOP RIGHT: Figure 5. Furnival Gardens is the site of the former Lower Mall in Hammersmith (destroyed in a World War II air raid) where Hatchett's home and laboratory were located, where he discovered columbium. The exact address and location are unknown.

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give the diagnostic Prussian blue color (iron ferrocyanide) and (2) its reaction with "tincture of galls" (gallic acid) to give a "rich purplish brown color" (iron gallate; this had been used as the basis for black ink for centuries).[3c]

Hatchett next turned to the white precipitate; his task was to ascertain if the product was a known substance. His choices were limited to (1) the twenty-two metals and earths designated by Lavoisier in his 1789 *Treatise*[2b] (Sb, Ag, As, Bi, Co, Cu, Sn, Fe, Mn, Hg, Mo, Ni, Au, Pt, Pb, W, Zn, Ca, Mg, Ba, Al, Si) and (2) the eight elements discovered since (Ti, U, Be, Y, Te, Zr, Cr, Sr). By series of established diagnostic tests, he was able to eliminate all of these possibilities: nonreactivity with nitric acid; orange precipitate with tincture of galls; nonreactivity with ammonia; and no formation of sulphuret [sulfide] when reacted with potassium sulfide. Hatchett deemed "it appeared proper that it should be distinguished by a peculiar name" since it originated from America—he gave it the name of *Columbium*.[3c] In 1805 the ore sample was formally named "columbite," originating from the "province of Massachusett."[4] [*sic*]

What was the geographical source of the columbium ore sample? In his announcement of columbium,[3c] Hatchett states that, according to its label, the sample was sent with various iron ores to Sir Hans Sloane by Mr. Winthrop of colonial Massachusetts. Hans Sloane (1660–1753) was the immediate successor to Sir Isaac Newton as president of the Royal Society in 1727. He developed the Chelsea Physic Garden [botanical gardens] (Figure 2) which Carl von Linné (Linnaeus), the great Swedish botanist, visited several times in the 1730s;[5a,d] Sloane is perhaps best known today as the one who introduced milk chocolate beverage to Great Britain.[5c] During his life Sloane amassed a huge collection of "curiosities" of natural history from about the world, with more than 100,000 items in his private museum including almost 10,000 mineral and stone specimens.[5a]

Which John Winthrop was specified on the label? There were five generations of Winthrops[6] living in Massachusetts during Colonial times, beginning with John Winthrop I (1587–1649)—the first governor of Massachusetts (1630–1642); he was the first to use the Biblical phrase "city on a hill" to idealize American culture)—and ending with "Professor" John Winthrop (1714–1779)—astronomer and professor at Harvard University, one of the last Colonial scientists. Three of the Winthrops were Fellows of the Royal Society of London. Most scholars agree[7] that the possibilities are limited to John Winthrop IV (1681–1747), who spent most of his life in England and his grandfather John Winthrop II (1606–1676), who developed iron works in Massachusetts and later became governor of the new colony of Connecticut.

Fortunately, a clearer story has since emerged with the subsequent research of Raymond Phineus Stearns (1904–1970),[8] an expert in Massachusetts colonial history and its scientific and commercial dealings with England.[6,9] The Royal Society of London, founded in 1660, encouraged exotic collections from around the world to be submitted. In 1669, John Winthrop II, a charter member of the Royal Society, sent the first gift of any size from the New World. Over the period of a century, a steady stream of "curiosities of nature" (e.g., "Shell of the Poquahauges . . . The English make pyes thereof: and of the Shell the Indians make ye following mony"[6]) were sent by him, his son Wait, his nephews, and then his grandson John Winthrop IV (1681–1747). In 1734 John Winthrop IV sent the largest single gift to the Royal Society Repository up to that time, consisting of "above six hundred curious specimens, chiefly of the Mineral Kingdom."[6] All these collections were sent from Boston, the main port of New England. Regrettably some of these were lost at sea, but those that arrived were received by the Repository of the Society and were carefully documented. There is no entry in any Winthrop list that identifies with Hatchett's columbite. (Note 1)

In 1779 the Society's Repository was closed out and all collections were donated in 1781 to the recently founded British Museum in Bloomsbury, which opened to the public in 1759 (still exists at same location, now much extended and modernized). These collections were added to the main body of collections at the British Museum donated by Hans Sloane. Unfortunately, through the years there had been so much commingling of Sloane's private collection and the Society Repository that it appeared "impossible now to separate the items and associate them accurately with their respective donors."[9]

The lists of "curiosities of nature" of John Winthrop II and his grandson John Winthrop IV are strikingly similar, with many duplications, with a literary form and terminology suggesting an earlier time.[9] Thus, it appears that John Winthrop IV's collections were assembled a century earlier,[9] and that he in his own name used collections of his grandfather to put himself in good stead with the Royal Society. With regard to the geographical origin of the columbite specimen, an almost unintelligible scrawl on the original label (recorded by Hatchett as *Nautmeage*), if read as *Nameauge* makes sense, because this was the common name for New London, Connecticut—where John Winthrop II resided as governor during the middle 1600s before the settlement was officially renamed New London in 1658.[6] Thus it is clear that the specimen was originally in the collection of Governor John Winthrop II. However, it is not known when the gift was made, by whose hand it was sent to England, and whether it was sent to the Royal Society or was given to Hans Sloane directly.

Unfortunately, it will never be known how the columbite sample was collected—but it was known that John Winthrop II made many mining and prospecting excursions,[6] including the area shown to be abundant in columbite, 25 km northwest (10 km southeast of Middletown[10]) (Figure 6). (Coincidentally, this was also near the collection site of the first helium-bearing uraninite in America.[2i]) Another possible source were friendly Indians, who would bring mineral samples they had collected from the Inland.[6]

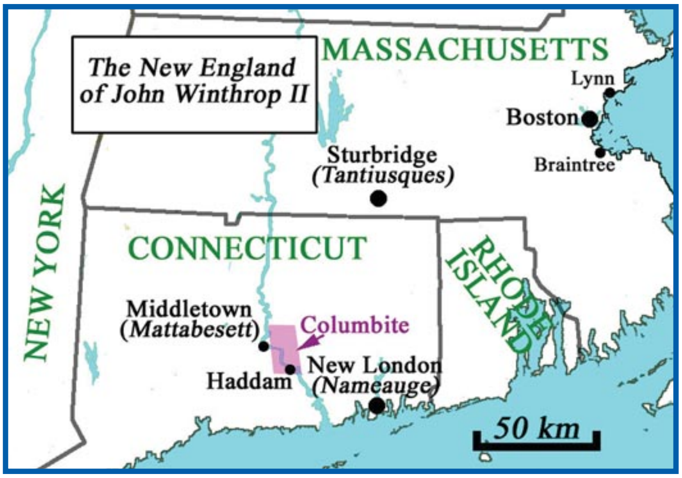

Figure 6. In 1631 John Winthrop II (1606–1676) joined his father, the founding governor, in the Massachusetts Bay Colony. John Winthrop II was a charter member of the Royal Society of London (1660) and was known as its "chief correspondent in New England."[6] During 1644–1646 he set up iron forgeries at Lynn and Braintree and a graphite ("black lead") mine at Tantiusques (today on the National Register of Historic Places, N42° 3.54 W72° 7.76). As governor of Connecticut (1657–1676), John Winthrop II lived in New London. His columbite specimen originated in the Middletown-Haddam area.[17a,b] Original Native American names of communities, used in the 1600s, are in parentheses.

Figure 7. Mount Clare, Hatchett's home during his middle years. Berzelius visited Hatchett here in 1812 and described his estate as an Italian villa with a very well-equipped laboratory nearby— which unfortunately Hatchett did not much utilize. Mount Clare is now owned by the University of Roehampton and is being remodeled for offices.

After the discovery of columbium. After about ten years of active chemical analysis, Hatchett moved on to other interests.[1] In 1809 he joined the Literary Club, where he enjoyed the company of Edward Gibbon, Adam Smith, Sir Walter Scott, Sir Humphry Davy, William Wollaston, and others. Upon his father's death in 1806, he received a sumptuous inheritance, and he moved into a new summer home in 1809, Mount Clare (Figure 7). Mount Clare had a neighboring laboratory building, but he rarely used it; many scientists rued the fact that Hatchett was no longer active in chemical analysis, but instead pursuing other artistic and worldly matters. In 1821 he repurchased and returned to his boyhood home, Belle Vue, (Figure 8) where this "jovial, cultured"[1] man could entertain and enjoy his "capital mansion" which commanded "beautiful views of the Thames and the distant Surrey Hills,"[5b] until his death at the age of 82, following his wife ten years earlier.

Figure 8. Charles Hatchett lived in Belle Vue in Chelsea in his early years, and then repurchased the site as his final home (1821–1844). Hatchett had amassed a huge collection of art and music treasures and he entertained frequently the higher society of London. At the time Hatchett lived here, this was a pastoral scene, where people could stroll leisurely down a wooded country lane; today the house is on a busy throughfare and the dwelling serves as an apartment complex. The Chelsea Physic Garden lies 850 meters northeast (right).

Figure 9. Ekeberg performed his work at the University of Uppsala. His tantalum was procured from yttrotantalite mined in Ytterby, Sweden[2f] (N59° 25.60 E18° 21.18) and from tantalite mined in Skogsböle, Finland. Nordenskiöld[18] described the Skogsböle site as "3/4 mile from the Kimito Church." A Swedish mile is equivalent to 10.7 km, placing the site 8.0 km west of the famous Kimito Church, dating from the 1200s. The Finnish Geological Survey furnished the information that this exact site (once a tin mine, now a feldspar quarry) is known: N60° 8.59 E22° 35.98. "Skogsböle" is an archaic Swedish name for "forest home."

Figure 10. Laboratorium Chemicum of the University of Uppsala, which was built in 1753, served the chemistry department until 1858. Here Torbern Bergman and Anders Ekeberg worked; tantalum was discovered here in 1802. Now the building is used by the Department of Hydrology. (Västra Ågatan 24, N59° 51.33 E17° 38.43). The present Kemicum (Chemistry Building) is 750 meters to the west, up a steep hill.

Anders Ekeberg[11] was a simple, quiet man immersed in the academia, both the sciences and literature (Figure 9). During his childhood a cold impaired his hearing, which handicapped him his entire life, and later a chemical explosion would take away sight in one eye. He matriculated in Uppsala University, studied at Greifswald (then Sweden-controlled northern Germany) and Berlin, and then returned to Uppsala in 1794. He worked up the ranks of the chemistry department (Figures 10, 11) and in 1799 was elected a member of the Swedish Academy of Sciences. He was involved in political activism, writing patriotic poetry about the Russo-Swedish conflict of 1788–1790.

In 1784 Ekeberg replaced the famous Torbern Olof Bergman (1735–1784), the director of the chemistry department at Uppsala. (Bergman was the mentor of Carl Wilhelm Scheele (1742–1786),[2c] who discovered Feuerluft (oxygen) and five other elements, as well as many organic substances, and hydrogen sulfide and hydrogen cyanide).[2c] Ekeberg introduced Lavoisier's antiphlogistic chemistry to Sweden, as well as Swedish names for oxygen, hydrogen, and nitrogen. His partner in this work was Pehr von Afzelius (1760–1843), who had visited Lavoisier during a tour of Europe. Afzelius was also the director of the M.D. dissertation of Jöns Jacob Berzelius (1779–1848)[2e] and introduced Berzelius to Ekeberg's new chemistry.[11]

Ekeberg was a skilled and careful analytical chemist, following the model of Klaproth whom he greatly admired.[11] After Bergman's passing, Ekeberg became the inspirational leader at Uppsala; Berzelius said his goal was to perform all analyses such that neither "Ekeberg nor Klaproth could correct his work."[11]

Ekeberg revisited the analysis of gadolinite, a mineral from Ytterby[2f] (Figure 12) from which Johan Gadolin (1760–1852) had discovered the first rare earth.[2f] Ekeberg was actually the person who gave the name "yttria," "Ytterjord" = "Ytter earth," to the new element in 1797.[2f] Ekeberg was trying to resolve the discrepancies between Klaproth[12] and Nicolas-Louis Vauquelin (1763–1829),[2a] the two foremost chemical analysts in the world—at the time of

Figure 11. Appearance of Uppsala during the time of Ekeberg, looking westward on the Fyrisan River, with the science building on the left side of the bank (over the shoulder, out of view). The Uppsala Cathedral (spires to the left) dates to the late thirteenth century. Further left is the dome of the Gustavianum. Courtesy, Museum Gustavianum, Uppsala, Sweden.

the discovery of yttrium in gadolinite ($Y_2FeBe_2Si_2O_{10}$), beryllium was unknown and Gadolin misidentified it as aluminum.[2f]

Ekeberg's careful work allowed him to recognize a new mineral from Ytterby.[12] The tests that Ekeberg used were similar to those used by Hatchett: he followed a series of analyses that finally narrowed down the list of possible known candidates as tin, tungsten, and titanium. But it couldn't be tin because tin oxide would have been easily reduced; nor could it be tungsten which is easily soluble in ammonia; nor could it be titanium, which becomes soluble in acid when fused with potassium carbonate. It must be a new element; he named it"tantalum." The mineral itself he called yttrotantalite (today known as $YTaO_4$).[12] Ekeberg further detected the new element in a mineral from Skogsböle, Kimito, Finland in a mineral he called "tantalite," which was composed of tantalum, iron, and manganese (today known as $(Fe,Mn)Ta_2O_6$).[12]

Comparison of columbium and tantalum—Wollaston's work. Subsequent chemical reports and reviews faithfully reported the results of Hatchett and Ekeberg, and chemical dictionaries recorded the two elements as separate entries for almost a decade. But many became suspicious, and William Hyde Wollaston (1766–1828), the discoverer of palladium and rhodium,[2h] procured samples of Ekeberg's tantalite and Hatchett's columbium for a direct comparison. In his classic paper in 1809, "On the Identity of Columbium and Tantalum,"Wollaston presented his study.[13]

Wollaston first treated the columbite and and the tantalite to remove the iron and the manganese to leave the white oxide residues (new element). Both the columbite and the tantalite oxides were insoluble in all three mineral acids (quite unusual for a metal), but both could be fused in potassium carbonate. Both the columbite and the tantalite oxides were soluble in oxalic acid, tartaric acid, or citric acid. Neither oxide gave a precipitate when reacted with infusion of galls (gallic acid), prussiate of potash (to give "Berlin blue," or ferric ferrocyanide), or hydrosulphuret of potash (potassium sulfide)—but when acid is added, each gave an orange precipitate with infusion of galls. The crystals of columbite and tantalite also appeared identical. Wollaston remarked,"I see no reason to doubt of their perfect agreement in all their chemical properties. . ."[13]

However, most perplexingly the specific gravities of the two minerals were quite different—columbite with 5.918 and tantalite with 7.953. Wollaston could not explain this difference: "The only chemical difference, by which this circumstance could be explained, would be the state of oxidation, which my experiments cannot appreciate; but it also may arise in part from actual cavities in the mass of columbite, and in part from the state or mode of aggregation."[13]

In the minds of the general scientific community, it was settled: columbium and tantalum were in fact the same element. But what would they call it?

Berzelius[2e] and Thomson[2d] disagree on nomenclature. In Thomas Thomson's (1773–1852) new *Annals of Philosophy*, he invited Berzelius to contribute his new treatise, "Essay on the Cause of Chemical Proportions."[14a] But Thomson made an editorial correction in the transcription and changed Berzelius' "tantalum" to "columbium."[14b] "Dr. Wollaston has shown that columbium and tantalum are the same metal. The first was discovered by Mr. Hatchett some years before tantalum was announced by Mr. Ekeberg. Mr. Hatchett's name, therefore . . . ought to be retained." Berzelius was swift to defend his idol Ekeberg:[14c] "I affirm that neither the properties of tantalum nor its oxide were known before the experiments of Ekeberg. . . . As for the name of the metal, Mr. Hatchett chose the name columbium from the place from which he supposed the specimen came; but this is not a good method of naming minerals. Besides, . . . [w]e are not sure the specimen came from America. . . . Perhaps the name tantalum (from the history of Tantalus) will appear still more appropriate, [because] . . . the metal . . . is not attacked by any acid. . . ." (It is to be noted that Berzelius did not object to Ekeberg's naming Gadolin's new element "Ytterjord" from the location where the gadolinite ore was found!)

Thomson sniffed,[14c] "I think *strontian* [named for a village in his native Scotland] just as good a name as *barytes* [Greek *heavy*]. . . I conceive too that Christopher Columbus has as good a claim to give his name to a metal as the King of Tantalus." Thomson continued to use "columbium" exclusively the rest of his life, as well as others of the British Isles, such as Sir Humphry Davy in his *Elements of Chemical Philosophy*.

The interchangeable use of columbium and tantalum continued for 32 years. The literature during this period can be confusing to the modern chemist. For example, one can read "Tantalite was first found in America, at Haddam, in Connecticut. . . ."[15] and "Haddam Columbite, when immersed in water, continued to give out minute bubbles of air for a considerable time, after which the specific gravity was much increased [thus more resembling tantalate]."[16] The lanthanide contraction[2i] was not yet known, and no one understood that two different elements could have such similar properties. ⬡

Next: Columbium (niobium) and tantalum are distinguished by Rose and Marignac, and the name niobium replaces columbium universally.

Figure 12. The Ytterby Mine (N59° 25.60 E18° 21.18) produced high quality quartz and feldspar for the porcelain trade in Britain and Poland. Today the mine is blocked and overgrown with vegetation. Ytterby means "outer village." Not only the first rare earths were discovered here, but also tantalum.

Notes.

1. Often cited in the literature is a publication where the editor suggests in the 1734 list of gifts,[17c] "A black mineral [entry 348], very heavy, from the inland parts of the country" may refer to the columbite sample, but Stearns showed that the Sloane numbers do not match[9] and that this cannot be the case.[9]

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