

## CHAPTER 1

### WHAT DID JOHAN GADOLIN ACTUALLY DO?

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#### 1.1. Johan Gadolin: a short biography and bibliography

Johan Gadolin (5 June 1760 - 15 August 1852) was born in Turku (Åbo) in Finland, which then was an integral part of Sweden. He first studied at the Royal Academy at Åbo in 1775 and later at Uppsala (1779-83), receiving a degree there in 1781. His thesis was called "*De analysi ferri*" and thesis advisor, Torbern Bergman (1738-84). He held professorial positions at Åbo from 1785 until his retirement in 1822.

Gadolin's papers are listed in the reprint and memorial volume by Hjelt and Tigerstedt (1910). Further details can be found in Asimov (1976), Enkvist (1972), Klinge et al. (1987), Mäkitie (1982), Pyykkö and Orama (1988), Sjöblom (1960a,b), Tigerstedt (1899) and Toivanen (1980).

#### 1.2. The discovery of the mineral

The Ytterby lanthanide mineral was discovered by Lieutenant Carl Axel Arrhenius and announced by Geijer (1788). The paper is short enough to be entirely reproduced on the cover of the "Abstracts and Programme" of the 2nd International Conference on Lanthanides and Actinides in Lisbon, Portugal in 1987. Geijer reports physical properties (black, asphalt- or coal-like colour, high density of 4.223, lack of magnetism of the stone or of the oxide, magnetism of the reduced metal). Preliminary experiments with a blowpipe and with calcination are mentioned. The only preliminary conclusion is a proposal that the sample might contain tungsten. A later paper on "Pitchblende" in Rinman's "Bergverks Lexicon" is quoted by Gadolin (1794, 1796).

## 1.3. Gadolin's analysis: The 1794 and 1796 papers

A rather more detailed analysis was reported by Gadolin (1794, 1796) in the nearly identical Swedish and German versions of the same paper, after he had obtained a small sample from "Captain Arrhenius". The organization and contents of the paper are as follows:

## §1.

Physical appearance: This mineral occurred in a red feldspar as rounded aggregates or as parallel sheets. The density (4.028 compared to water, see Gadolin (1792)), the grey-green colour of the ground powder and an idea of the hardness are given.

## §2.

Gadolin managed to melt, with difficulty, the stone in a blow-pipe into a black, bubbly slag, in contrast to Geijer, who failed to achieve this, and Rinman, who obtained a yellow glass. Melts were also obtained with sodium hydroxide [Soda-Alkali] borax and potassium hydrogen tartrate ["*Sal microcosmicus*" (Swe.)/ "*Weinsteinsalz*" (Ger.)]. A mixture of the ground stone with two parts of saltpetre did not explode in a glowing crucible. Leaching with water and drying yielded a brown powder of the original weight. The metallic residue of Geijer and Arrhenius is quoted.

## §3.

A) A ground sample of the stone was dissolved in concentrated nitric acid, giving a white, insoluble powder in a greenish solution. Evaporation of water gave a jelly, redissolved by water.

B) The brown powder, obtained by melting in saltpetre, behaved similarly.

C) Solutions in hydrochloric acid also gave a greenish solution and an insoluble powder. Some heat and a little gas were generated. A smell, not unlike the "hepatic", was detected. Strong acid gave a thick, oily solution. The final colour was brown-yellow.

D) Hydrochloric acid also attacked larger pieces of the stone, giving a white remnant stone and a similar solution.

## §4.

The insoluble residue was dried and had a weight of about 1/3rd of the original. It could not be melted in a blow-pipe, not even with feldspar. With sodium carbonate a clear glass was obtained, with evolution of gas. On cooling, the glass became milky. Saturation with saltpetre gave a white pearl of enamel. Borax gave a clear glass. *Sal microcosmicus* gave no reaction.

It consequently follows that the part, insoluble in acids, mainly contains silica, (underlined by translators), but that it also contains something else less capable of reacting with alkali carbonate. Undoubtedly, some feldspar was present. Whether a metal oxide, insoluble in acids, could also be present was investigated as follows: 1 part of the insoluble residue was mixed with 3 parts of crystalline sodium carbonate and calcined in a crucible for one hour. A spume, that was mostly soluble in water, resulted. The insoluble part, rinsed and dried, had 1/5th of the weight of the entire residue. From it, sulphuric acid could extract a slight amount of aluminium oxide.

The remnant was mixed with 3 parts of sodium carbonate, calcined and dissolved in water. The residue that remained undissolved had about 1/20th of the mass of the original residue, but it behaved similar to the way it had before alkali melting; i.e. with sodium carbonate it melted, with gas evolution, into a pearl which was clear when warm but opaque when cool. Once, a small silvery metal fragment was observed in the pearl. A slight addition of copper oxide gave the glass a ruby-red colour. This suggests that the residue may have contained some tin oxide. No tin layer could be obtained on an iron wire inserted into the glass, however.

After calcination with sodium carbonate the water-soluble part formed a jelly with hydrochloric acid, behaving just like pure silica [water glass].

## §5.

A) The solution in hydrochloric or nitric acid (§3), containing 2/3rd of the weight of the earth, gave in pure potash solution first a brownish, then a white precipitate which, rinsed and dried in warm air, weighed somewhat more than the entire earth before dissolution.

B) With ammonia, the same solution gave a grey precipitate, turning dark brown when heated. After addition of a slight excess of ammonia, all earth was separated from solution, and no further precipitation resulted with alkali [*feuerfeste Laugensalz*]. Hence these solutions contained no observable traces of tungsten oxide [*Schwererde*] nor of calcium or magnesium oxide; this conclusion will shortly be confirmed. The precipitate with ammonia had, after rinsing and drying, somewhat more than the mass of the earth. Its mass was reduced through calcination to 2/3rd of the original; so was that of the precipitate (A).

Both precipitates behaved similarly in blowpipes. They darkened during heating, hardened at higher temperatures and became slaggy on the surface, but did not melt. With borax they yielded a clear, greenish or black glass, depending on the proportions, and with *Sal microcosmicus* a clear glass, containing white particles above the phase separation. These did not dissolve by melting with sodium hydroxide [*Alkali Sodae*]. The white precipitate, finally obtained by potash solution, collected and dried became bleak red-grey as it dried. By blow-pipe in the presence of borax it gave a clearer glass than in the outer flame obtained a hyacinth colour; it finally became opaque when blown for a long time at the extremity of the flame. It became clear again in the blue flame.

C) Macquer's "*Blutlauge*" ( $K_4[Fe(CN)_6]$ ) separated from the mentioned solutions a large, blue precipitate in which a slight amount of white powder could be seen. Thus 100 parts of the powdered earth yielded 41 parts of precipitate. This corresponds to 12 parts iron oxide if a pure calcined iron oxide is dissolved in the same acid and precipitated by the same "*Blutlauge*".

With potassium hydroxide the clear solution that was not precipitated by  $K_4[Fe(CN)_6]$ , gave a white precipitate that, after rinsing and drying in warm air, weighed 87 parts. This was partially dissolved by concentrated potassium hydroxide [caustic vegetable alkali]. Addition of vitriolic acid to the obtained alkaline solution gave a white precipitate, with a cubic crystal habit, weighing 29 parts. It was soluble in vitriolic acid and gave, by evaporation, crystals of alum. The part not dissolved by alkaline potash solution was an unknown earth that shall be described more closely below. After rinsing and drying, its mass was 38 parts.

Using the weights after heat treatment, the various constituents of 100 parts of the black earth contain approximately:

31 parts silica
19 parts aluminium oxide
12 parts iron oxide
<u>38</u> parts of an unknown earth.
100

Sometimes the undissolved silica appeared to contain some tin oxide, which probably formed the white particles in the  $K_4[Fe(CN)_6]$  precipitate (see next section). The blowpipe behaviour also suggested some manganese dioxide.

#### §6.

The brown, dried precipitate of §5A was analysed further as follows:

A) It could be slowly dissolved in distilled, weak acetic acid, requiring 120 parts thereof. Some brown-black powder remained; it consisted of iron oxide, with some aluminium oxide.

B) Dilute sulphuric acid dissolved it entirely, developing initially both gas and heat. The brown powder in the bottom disappeared by gentle heating. The solution was pale green. A piece of blank iron, immersed in this solution, was attacked by the remaining acid. No metallic precipitate [consisting of nobler metals] appeared on it. An immersed copper sheet did not change its colour either. If both iron and copper were immersed, a thin white metal layer appeared on the latter.

C) Nitric acid similarly dissolved it, with the evolution of gas, into a clear solution which, by evaporation, became viscous and later formed a jelly. By further heating it foamed, emitting red-brown fumes, and finally became a dark-brown residue containing white particles. The same procedure was repeated with the same amount of nitric acid (2 parts of acid to one part of precipitate). Finally, 2 parts nitric acid and 4 parts water were added to the dark-brown powder. The solution was boiled and diluted with 10 parts water. Upon filtration the solution left a rust-brown powder, 4 per cent by weight of the original precipitate. The filtered solution gave with potash solution first a white precipitate which dissolved again as the acid approached neutrality. When further alkali was added, the solution became yellow, turning later dark-brown, opaque, and precipitated a dark-brown powder, increasing upon boiling. Separated and dried, these black, cubic crystals weighed 16 per cent of the original material.

The filtrate was clear, despite containing dissolved iron oxide. With potash solution it gave a white, slightly reddish precipitate, weighing 80 per cent of the original material when rinsed and dried. It was dissolved in nitric acid, precipitated again with potassium hydroxide and rinsed with water. While it was still wet, concentrated potassium hydroxide was added. Upon heating it leached 20 per cent of its substance which behaved as pure aluminium oxide. (This percentage is slightly below that in the previous paragraph, due to previous coprecipitation with iron oxide).

The remaining part of the powder that was not dissolved by the potassium hydroxide had properties similar to the unknown earth of §5.C, except that it still contained some iron oxide, making it slightly reddish. It was purified by dissolution in acid and precipitation by  $K_4[Fe(CN)_6]$ .

#### §7.

The unknown earth had the following properties: Even at the highest temperatures obtained with the blow-pipe, it remained white and did not melt [the melting point of  $Y_2O_3$  is  $2410^\circ C$ ].

Borax dissolved it into a clear, colourless glass.

With sodium ammonium hydrogen phosphate it yielded a glass, which remained colourless in the blue flame of the blowpipe but became milky in the outer flame.

With boric acid it gave a white enamel.

It did not dissolve in sodium hydroxide upon melting.

High-temperature treatment in a crucible placed within a coal-oven did not give any change under conditions which reduced iron. Melting with borax under the same conditions also failed to yield any metal.

In wet chemistry this earth easily dissolved in most acids giving clear, colourless solutions.

Its *sulphuric acid* solution did not yield bigger crystals. During evaporation a white powder was obtained. Its taste, after rinsing, was sweet-sour. If the solution was fully dried, a white powder was obtained that, in its appearance and slight solubility, rather resembled selenite but, in its taste, alum. An excess of sulphuric acid readily dissolved it in water.

With *nitric acid* a clear solution was obtained. Upon evaporation it formed a jelly and could not be brought to crystallization.

A solution in *hydrochloric acid* showed no tendency to crystallize either, instead becoming viscous. From all these solutions the earth could be precipitated by ammonia. The precipitate was insoluble in alkaline solutions.

*Carbon dioxide* reacted with this earth in the sense that the precipitate obtained with alkali carbonates evolved gas when treated by acids. This precipitate was only slightly soluble in carbonated water (1 part in 7000).

*Phosphoric acid*, obtained from phosphorus by slowly burning it in air, did not precipitate the earth from its solutions in other acids. Phosphoric acid, applied on the dry powder, evolved gas and dissolved a small amount. The solution had a sour, contracting (astringent) taste, and became gelatinous under evaporation. If the phosphoric acid was saturated with the earth, the substance was insoluble in water. After drying, this saturated compound weighed more than twice as much as the earth. It did not melt in the blow-pipe and gave with borax a yellowish glass, becoming opaque in the outer flame.

The reaction with *boric acid* took place through double decomposition. A borax solution was added to a solution of the earth in another acid (one part of earth in 7 parts diluted sulphuric acid: 3 parts of water and 1 part of concentrated acid). The result was mixed in 100 parts water. Into this, the borax solution was gradually added. This gave a temporary opalescence disappearing again in acid solution. Later a white

["gelb/hvit"] precipitate appeared, ceasing after 5 parts of borax had been added, the solution still remaining slightly acid by a litmus test. The obtained precipitate, weighing about one-half of the dissolved earth, melted in blow-pipe to a milky pearl. With added sodium ammonium hydrogen phosphate the pearl became white and opaque. With borax a half-clear glass was obtained. After extended blowing it became completely clear.

Under slow evaporation the clear solution first gave a number of clear, needle-shaped crystals, then some larger octahedral clear crystals and after that clear boric acid and Glauber's salt [ $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ]. The needle-shaped crystals had limited solubility in water, little taste and disintegrated when heated, melting at high temperatures to a white enamel which finally clarified into a glass pearl. The octahedral crystals were more easily soluble, had a sweet taste and left a feeling of heat on the tongue. They contained plenty of water of crystallization, causing foaming in the blow-pipe. At higher temperatures the salt became white and opaque, melting afterwards to a clear glass.

*Oxalic acid* precipitated the earth from its solution in all other acids, unless an excess of that acid was present. Thus the earth was entirely precipitated from its solution in sulphuric acid when oxalic acid, saturated with potassium hydroxide, was added. The precipitate was a white powder that was carbonated in the blow-pipe, burned with a fiery flame, then becoming white and behaving like the original earth. A part of this precipitate was calcined in a crucible whereby it lost half of its original weight. The heated powder, dissolved in sulphuric acid, gave by slow evaporation clear, irregular crystals. When all superfluous sulphuric acid had been dried away, and the dry residue dissolved in water, renewed evaporation gave sharp, rhombic crystals, having a sweet taste and contracting the tongue. These crystals were stable in air. In the blow-pipe they disintegrated, forming thin sheets that did not melt alone at high temperatures and gave a milky glass with borax. One part of these clear crystals required 22 parts of water for dissolution at normal temperature. Because such crystals were not obtained when only the earth was dissolved in sulphuric acid, it appears that some part of the organic acid had survived the calcination.

The earth was similarly precipitated by a saturated alkaline solution of oxalic acid from its solutions in hydrochloric acid. As, however, this solution had a strong excess of acid, complete precipitation of the earth was not obtained. After evaporation the mother liquor gave under evaporation clear, rhombohedral crystals resembling gypsum and consisting of parallel, rhombohedral sheets. These were dissolved in the blow-pipe in their own water of crystallization. At higher temperatures they were carbonated and transformed into porous carbon, melting finally into a white, opaque pearl, having a caustic, alkaline taste. Hence this salt seems to consist of the earth, combined with oxalic acid and potassium hydroxide. It dissolved quite easily in warm water but required, at average temperatures, 45 parts water to one part salt. Addition of *tartaric acid* to a solution of the earth in hydrochloric acid gave no precipitate. If, however, the extra acid had been neutralized by alkali and the added tartaric acid was likewise saturated with potash, a large white precipitate resulted. It seemed to be more soluble in pure water than the oxalic compound.

*Acetic acid* dissolved the earth easily, with evolution of gas.



Johan Gadolin (Courtesy of the National Museum of Finland)

## Conclusion

"These properties suggest that this earth has similarities with alum and with CaO, but also dissimilarities from both of these, as well as other known earths, whence it should be included among the simple earths, unless the experiments lead to a conclusion that it is composed of several substances. I do not yet dare to claim such a discovery, both because my limited supply of the black mineral has not permitted all desired experiments and because science would benefit more, if the new earths recently described by chemists could be separated into simpler constituents, than in the case that their number is further increased" (Gadolin, 1794, 1796).

## 1.4. Gadolin's letter to Wilcke

The manuscript of Gadolin (1794) was mailed to Professor J. C. Wilke. In the covering letter, dated 22 May 1794 (in Swedish, reprinted on p. xci of Hjelt and Tigerstedt (1910)), Gadolin thanks Cap. Arrhenius for providing a few years ago the sample of the black, shiny stone, found among the red feldspar in the Ytterby mine.

"Had I had more of the stone I would not have considered my experiments finished".

"It is not without great trepidation, I dare speak of a new earth, because they are right now becoming far too numerous, unless one rapidly finds a way to analyse them further. However it seems to me that mine should have some similarity with Klaproth's zirconia"...

"Should the Ytterby earth be identical with zirconia, much would be gained, for it seems to me rather fatal if each of the new earths should only be found at one site or in one mineral".

As a curiosity we note the Editor's foot-note on p. 313 of Gadolin (1796): The author's submission letter was dated 3 July 1794 but reached Crell on 26 March 1796, perhaps because the letter had apparently been given to a traveller. "Meanwhile, the paper has lost nothing but, would it be written now, it should contain some clarifications due to Prof. Klaproth's excellent contributions".

## 1.5. The first confirmation by Ekeberg

The first confirmation of Gadolin's analysis was published by Ekeberg (1797), who had obtained for this purpose from Captain Arrhenius a larger sample without feldspar. This diminished the amount of silica and aluminium oxide obtained. His result was:

Silica	25 parts
Iron oxide	18 parts
Aluminium oxide	4½ parts
The new earth	<u>47½ parts</u>
	95 parts
Missing	5 parts.

[In retrospect, the mineral "gadolinite" is  $\text{FeBe}_2\text{Y}_2\text{Si}_2\text{O}_{10}$ . The beryllium was confused with aluminium.]

The properties of the new earth fully agreed with those, reported by Gadolin. Ekeberg further mentions the sweet taste, "almost like that of lead compounds, but not so repulsive [*äcklig*] but rather contracting [*sträv och adstringent*]. The acetate was to my taste just as sweet as lead sugar [lead (II) acetate]."

Beautiful, air-stable crystals of the sulphate and acetate were obtained and described in detail. Solubility in arsenic acid and the slight solubility of the arsenate were discussed. Ekeberg gives a clear summary of the differences between the new earth and the previous ones. He proposed the Swedish and Latin names of "Ytterjord" and "Yttria", respectively, for the earth and "Yttersten" for the mineral. Finally he suggests that yttria, whose solutions have such an original taste, could also eventually be medically potent.

## 1.6. Acknowledgement

We thank Margareta Jorpes-Friman, Åbo Akademi Library, for providing us with copies of the original articles.

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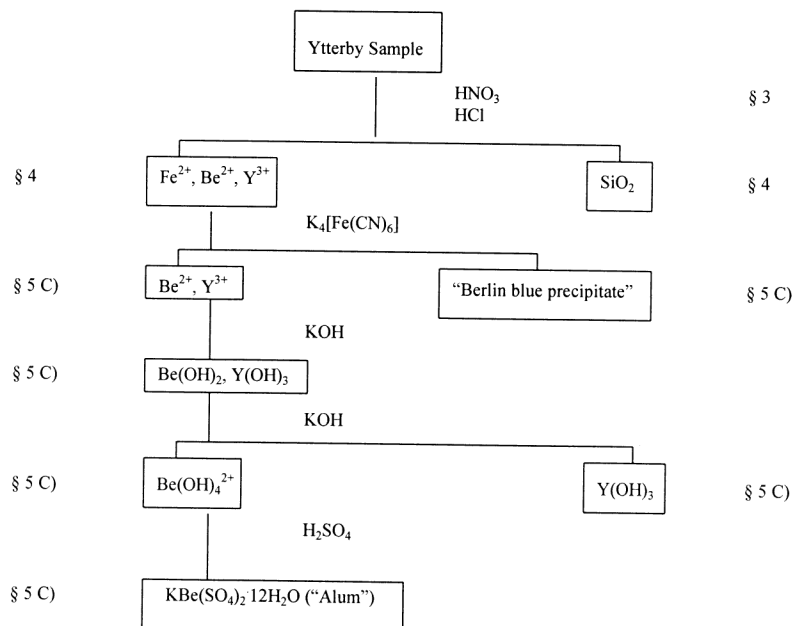
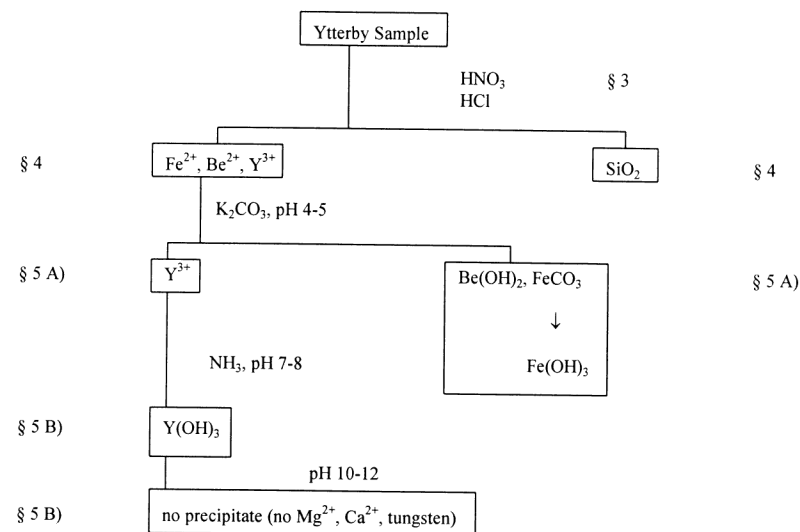
## Appendix 1- Historical nomenclature

Original Name Swedish/German	Page Swedish/German	Present English Name	Formula	Reference
Acetosellsyra/Acetosselsäure	153/327	Oxalic acid	$C_2H_2O_4$	d
Alkali/Laugensalz	142/318	Sodium or potassium carbonate	$Na_2CO_3, K_2CO_3$	a
Alkali aëratum/Luftgesäuertes	151/325	Sodium or potassium carbonate		b
Alkali				
Alkali minerale/Mineralalkali	143/318	Sodium carbonate	$Na_2CO_3$	c
Alun/Alaun	143/318	Alum	$K_2Al_2(SO_4)_4 \cdot 24H_2O$	c
Alunjord/Alaunerde	143/318	Aluminium oxide	$Al_2O_3$	b
Blodlut/Blutlauge	145/320	Potassium ferrocyanide	$K_4[Fe(CN)_6]$	d
Caustik pottaska/Kaustischer	146/321	Potassium hydroxide	KOH	b
Laugensalz				
Caustict flygtigt alkali/	144/319	Ammonia (concentrated)	$NH_3$	b
Kaustischer flüchtiger Laugensalz				
Flyktigt alkali/Flüchtiges Alkali/	144/319	Ammonia	$NH_3$	b
Volatile alkali				
Flusspat/Flusspat	141/317	Calcium fluoride	$CaF_2$	d
Fätspat/Feldspat	137/313	Potassium aluminium silicate	$KAlSi_3O_8$	d
Järnkalk/Eisenkalk	146/321	Iron oxide	$Fe_2O_3$	b
Kiseljord/Kieselerde	144/319	Silica	$SiO_2$	b
Luftsyra/Luftsaure	151/325	Sodium ammonium hydrogen phosphate	$Na(NH_4)HPO_4 \cdot 4H_2O$	b
Sal microcosmicus/Harnsalz	150/324			
Sal microcosmicus/Weinsteinsalz	139/315	Potassium hydrogen tartrate	$KC_4O_6H_5$	b
Sockersyra/Zuckersäure	153/327	Oxalic acid	$C_2H_2O_4$	b
Vegetabiliskt alkali/Gewächssalkali	145/320	Potassium hydroxide	KOH	b

References:

- Holleman-Wiberg (1976)
- Pearce (1987)
- Hagg (1963)
- Römpf (1966)

## Appendix 2 - The analysis schemes used



## CHAPTER 2

## THE DISCOVERY OF CERIUM - A FASCINATING STORY

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## 2.1. Introduction

The discovery of a new element is not an isolated event. Several small consecutive observations often lead to new presentations of a problem. When the time is ripe and the different observations combine with new knowledge, all the bits and pieces fit together and a beautiful new discovery is made.

The discovery of cerium is a brilliant example of the activities which took place in chemical laboratories in Sweden at the beginning of the 19th century. It illuminates the discussion and the choice of problems at that time, the uncertainty of the analytical techniques and the difficulties in the interpretation of the results. What directed the chemist's choice of a problem? The tradition within mining established almost immediately the field of mineral analysis, although it had become very popular to investigate the effects of the recently described voltaic pile. The high activity surrounding pneumatic chemistry around the 1770s and 1780s had decreased. A reason for the analysis of a certain mineral could be a divergent physical property such as density or crystal form, which could not be explained by the knowledge of that time. Seldom a practical problem influenced the direction of mineral analysis. Yet scientists often discussed solutions to technical problems associated with the handling of slag, the construction of furnaces etc, and several journeys were made by Swedish mining engineers to study mining mechanics and the iron industry abroad.

Axel Fredrik Cronstedt, chemist and assayer, had described the discovery of two minerals with an exceptionally high density in the Transactions of the Swedish Academy of Sciences in 1751.<sup>1</sup> They were named tungsten ("heavy stone") and had been found at Bisberg near Säter and Nya Bastnäs or St Görans mine in the neighbourhood of Riddarhyttan. After preliminary experiments, Cronstedt suggested that the mineral from Riddarhyttan should be tested for an assumed content of zinc and