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Vanadium to dubnium: from confusion through clarity to complexity

Norman N. Greenwood*

School of Chemistry, The University of Leeds, Leeds LS2 9JT, UK

Abstract

The tangled history of the discovery and naming of the three elements vanadium, niobium and tantalum some 200 years ago is reviewed. Similar controversies attended the much more recent synthesis and naming of the heaviest element of the group, dubnium. Many lessons can be learned from these disputes which are still very relevant today. There follows a brief summary of the physical, chemical and biochemical properties of the elements and an assessment of their current significance.

Keywords: Vanadium; Niobium; Tantalum; Dubnium

1. Introduction

Centenaries are rare and are worth celebrating because they link the past to the present and invite a purposeful consideration of the future. Bicentenaries are even more rare, especially in chemistry, and provide an even longer perspective. But the triple bicentenary that we are celebrating at this International Symposium is unique in the annals of our subject because the history of all three of the naturally occurring elements in Group 5 of the periodic table began almost simultaneously in 1801–1802. We can learn much from a consideration of the confusions and errors that bedevilled the early decades of this saga. In our own times, the remarkable story of the heaviest member of the group, the artificially produced highly radioactive element dubnium, reminds us that confusion and error are not the sole prerogative of the 19th century but can still persist today. There are many lessons still to be learned.

2. The discovery of vanadium, niobium and tantalum

The complicated story of the successive discovery, rediscovery and naming of the three elements now known as vanadium, niobium and tantalum has often been told and has been scrupulously documented in detail in Gmelin [1] and elsewhere [2]. The element with atomic number 23 that we now call *vanadium* was first discovered in a Mexican lead ore by Andrés Manuel del Río in 1801. Note the date: this was before Dalton's atomic theory (1803–1808), before Mendeleev's periodic table (1869) and more than a century before Moseley's atomic numbers (1913). Things were very different in those days. del Río (1764–1849) was born in Madrid, graduated from the University of Alcalá de Henares and the Mining Academy of Almadén and then studied further in France, England and the renowned Royal School of Mines in Freiberg, Saxony, where he became a close friend of the celebrated German naturalist and traveller Baron Alexander von Humboldt who was a fellow student. In 1794, whilst not yet 30 years

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* Tel.: +44-113-343-6406; fax: +44-113-343-6565.
E-mail address: n.n.greenwood@chem.leeds.ac.uk (N.N. Greenwood).

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old, he was appointed as professor of mineralogy at the School of Mines in Mexico, recently founded by Don Fausto d'Elhuyar. (There is no need to remind an audience in Spain that the d'Elhuyar brothers had discovered tungsten (formerly known as wolfram) in 1783 just 11 years earlier.) del Río's journey from Cadiz to Veracruz in Mexico took 11 weeks to complete and this isolation from the centres of European research played an important role in subsequent events. During some work on a brown lead mineral from Zimapán in Hidalgo Province, Central Mexico, he isolated compounds of a new element which he first called *panchromium* because of the varied colours of its salts, but later changed this name to *erythronium* because of the red colours formed on heating or treatment with acids. There is some doubt about the date (1801 or 1802), because the contemporary records of the Research Institute can no longer be found, but the brown lead mineral from Zimapán was almost certainly what we now know as vanadinite, $3Pb_3(VO_4)_2 \cdot PbCl_2$. The earliest published account still extant is in Gazeta de Mexico No. 22/23 of 12 November 1802 [1]. del Río gave several specimens of the brown lead ore to his friend von Humboldt who visited Mexico in 1803 and he also sent some to the Institut de France, but a more detailed account of his work was lost in a shipwreck. Since the properties of erythronium appeared to resemble closely those of chromium, an element that had just recently been discovered by the French chemist Vauquelin in 1794, del Río lost confidence in the importance of his discovery and concluded that his supposed new element might simply be chromium. His confidence was further shaken in 1805 when his mineral was examined in Paris by Vauquelin's friend, H.V. Collet-Descotils, who concluded (erroneously, we now know) that the supposed new metal was indeed chromium and that the brown mineral was a basic lead chromate. It is clear to us today, though unfortunately not to del Río 200 years ago, that chromium (element 24) is the horizontal neighbour of vanadium (element 23) in the periodic table and also has many coloured compounds. del Río withdrew his claim for a new element.

Let us now "fast-forward" to 1830 when the Swedish chemist Nils Gabriel Sefström (1787–1845) discovered an apparently new element in iron from the Taberg Mine in Småland. Because of the richness and variety of colours of its compounds he called it vanadium after Vanadis, the Scandinavian goddess of beauty. To cut a long story short [1,2], further semi-collaborative work in 1831 by Sefström, by his mentor in Sweden J.J. Berzelius, and by the young German chemist Friedrich Wöhler (1800–1882) established the chemical uniqueness of vanadium and its identity with del Río's "erythronium". So, who discovered vanadium: del Río, who first discovered the new element "erythronium" in Mexico but did not conclusively distinguish it from chromium and then withdrew his claim? Or Sefström who discovered and named the element vanadium in samples of iron smelted from a mine in Sweden? Or Wöhler who showed that vanadium was the same element as the already known "erythronium"?

Now back to 1801 to unravel the history of our next element *niobium*. The element was announced by Charles Hatchett (1765–1847) in a paper read to the Royal Society in London on 26 November 1801 and published the following year [4]. Hatchett was a young man of independent means who had been elected a Fellow of the Royal Society in 1797 at the early age of 32 years. He had been examining some mineral samples that had been lying unstudied in the British Museum since at least 1753 and which may have originally been presented to the Royal Society as early as 27 June 1734 by John Winthrop of Massachusetts at the time of his own election as a Fellow [2,5]. The precise origin of the specimens is uncertain because the native name of the place where they were collected, Nautneague, no longer existed by the turn of the century: it may have been in Massachusetts or possibly in Connecticut. Note that the original transfer from the New World to England was made several decades before the American War of Independence and the foundation of the United States of America. In any event, Hatchett was able to isolate the oxide of the new element and unequivocally establish its chemical identity. He proposed to call the element *columbium* and the mineral columbite in honour of the poetic name of its land of origin. (We might parenthetically insert here the question, "Who actually discovered America"? Certainly not Christopher Columbus, who didn't know where he was going when he set off, didn't know where he was when he arrived, and didn't know where he had been when he returned. He certainly never set eyes on the American continent, let alone feet.)

Meanwhile, in Sweden, Anders Gustaf Ekeberg (1767–1813) was studying some Finnish minerals and in the following year, 1802, claimed to have identified a new element that he called *tantalum*, partly, as he said, “in allusion to its incapacity when immersed in acid, to absorb any and be saturated”. In modern terminology he meant that he had difficulty in dissolving the mineral in acids. (The classical allusion to Greek mythology might also escape a modern audience: it refers to Tantalus the mythical king of Phrygia, son of the god Zeus and a nymph, who was condemned for revealing the secrets of the gods to mankind—perhaps one of the earliest recorded examples of pillow-talk and the still rampant habit of “kiss-and-tell” indiscretions; one of his punishments was being made to stand in Tartarus up to his chin in water, which constantly receded as he stooped to drink.) Ekeberg communicated his findings to the French journal *Annales de Chemie* in 1802 and it is interesting to note from the title page of the Journal that the editors include Citizen Guyton and Citizens Berthollet and Vauquelin. We are further reminded of the stirring post-revolutionary times by the absurd dating system, “30 Messidor Year 10” which translates to 18 July 1802, as the Royal Society Librarian has helpfully indicated on the copy I consulted.

It was subsequently thought that the two elements columbium and tantalum were one and the same and this view persisted until at least 1844 when Heinrich Rose (1795–1864) examined a sample of columbite and showed that two distinct elements were involved. One was Ekeberg’s tantalum and the other he called niobium (after Niobe, the daughter of Tantalus). Despite the chronological precedence of Hatchett’s name columbium, the International Union of Pure and Applied Chemistry (IUPAC) finally adopted the name niobium for element 41 in 1950 after more than 100 years of controversy though, as you will know, the name columbium is still widely used by metallurgists and by parts of the US chemical industry. The great physical and chemical similarity of niobium and tantalum, deriving from the effects of the lanthanide contraction, is well known to you all and this similarity bedevilled earlier attempts to prepare pure samples of the individual metals and their compounds until well into the 20th century. For example, the mineral columbite is now formulated as $(Fe, Mn)(Nb, Ta)_2O_6$ and contains 47–78% Nb and trace-34% Ta depending on the source. As with the lanthanide elements, this great similarity also lead to many spurious claims for new elements among which can be mentioned H. Rose’s “pelopium” (Pelops was the brother of Niobe), von Kobell’s “dianium” (after the Greek goddess of the hunt, Diana), R. Hermann’s “ilmenium” (from the Ilmen Mountains, Urals) and his “neptunium” (from a Connecticut mineral) [1]. Even “euxenium” made a brief illusory appearance in the 19th century literature.

3. The synthesis of dubnium

Element 105, now named dubnium, does not exist in nature. Attempts to synthesise it began in the late 1960s and by 1970 serious claims to have succeeded were being made by rival groups working in Dubna, USSR, and Berkeley, CA. As with the preceding element 104, now named rutherfordium, which was also claimed by the two groups, an acrimonious controversy ensued and the matter was only resolved after an extensive investigation by an International Group set up jointly by IUPAP and IUPAC in 1987. I was privileged to be one of the two chemists on this Transfermium Working Group (TWG) and our final report was published in 1993 [6]. It is instructive to see why the controversy lasted so long and became so bitter but first let me indicate the nuclear reactions that were employed in the synthesis and the nuclear properties of the new element.

The Dubna team, under Flerov and Oganessian, working at the Joint Institute for Nuclear Research (JINR), bombarded americium-243 with neon-22: ${}^{243}_{95}\mathrm{Am}({}^{22}_{10}\mathrm{Ne}, 4n){}^{261}_{105}\mathrm{Db}$ and ${}^{243}_{95}\mathrm{Am}({}^{22}_{10}\mathrm{Ne}, 5n){}^{260}_{105}\mathrm{Db}$. The Berkeley team, under A. Ghiorso, used the reaction: ${}^{249}_{98}\mathrm{Cf}({}^{15}_{7}\mathrm{N}, 4n){}^{260}_{105}\mathrm{Db}$.

As the total yields of these early experiments were only a few atoms and the half-lives of the dubnium-260 and dubnium-261 isotopes were only 1.5 and 1.8 s, respectively, there was great difficulty in unambiguously characterising the products and demonstrating that the activities observed signalled the presence of a new element. Other nuclear syntheses followed and at least nine isotopes of element 105 are now known of which dubnium-262 has the longest half-life (34 s). One unfortunate and persisting consequence of the prolonged controversy is that element 105 has been referred to in the literature by several unofficial names. The most prominent of these are “hahnium”, which appears in much of the American literature and “nielsbohrium” which was originally proposed by the Dubna group: neither is now recommended by IUPAC as the name for element 105, though the recommended name for element 107 is bohrium.

In one sense it does not matter to science in general who it was who discovered a particular element, though to the scientists concerned priority is understandably often of considerable importance. Personal pride, institutional pride and even national pride become involved and, when experiments involve huge experimental costs over long periods of time, reputations and continued funding may well also be affected. In trying to unravel priorities it soon became clear to the TWG [6] that

> “The discovery of a new element is by no means always black-and-white in the sense that it can be unequivocally asserted that a new element was discovered with the required certainty, by a certain group, using a certain method on a certain date. Sometimes this is the case, and this is what is popularly thought of as a “discovery”. Perhaps more often, however, the situation is one in which data, accumulated over a period of time, perhaps of years, perhaps in two or more laboratories, gradually bring the scientific community to the conviction that indeed the existence of a new element has been established.”

As a result of these considerations the concept of “discovery profiles” was developed since the demand for an absolute assignment of priority in each case would imply attachment to outmoded concepts of the nature of discovery, particularly in those cases that concerned, rather, the *synthesis and characterisation* of the new element as opposed to its *discovery*. In the specific case of element 105, the TWG concluded that independent work reported in 1970 from Berkeley and from Dubna was essentially contemporaneous and equally convincing, and that credit for the discovery should be shared [6]. Following further lengthy consultations world-wide and deliberations in the appropriate committees, IUPAC finally recommended in 1997 that the approved name for element 105 was dubnium [7] and this has been widely accepted by the scientific community.

The synthesis of dubnium involved outstanding feats of experimental virtuosity and a full description of the work can be found in the papers cited in [6]. There is, unfortunately, no time to recount here the details of the elegant and sophisticated techniques developed by the rival groups, but it is clear from a rereading of the original papers after an interval of 30 years that much of the ensuing controversy could have been avoided if the temptation had been resisted to report work in progress rather than to wait for a complete and definitive characterisation of the new element before claiming and naming it. The subsequent heroic investigations of the chemical properties of dubnium on an atom-by-atom basis as it was being synthesised and isolated will be discussed towards the end of the lecture.

4. Atomic and physical properties of vanadium, niobium and tantalum

Having described the progression from confusion to clarity in establishing the existence of the three elements vanadium, niobium and tantalum during the first third of the 19th century it is now appropriate to summarise their most important atomic and physical properties before going on to discuss the increasing complexity of their chemical compounds.

As members of Group 5 of the periodic table, the elements all have odd (rather than even) atomic numbers. There are three immediate consequences of this. First, their cosmic and terrestrial abundances are an order of magnitude less than those of their immediate horizontal neighbours in Groups 4 (Ti, Zr, Hf) and 6 (Cr, Mo, W) [8]. Second, they each have only one stable naturally occurring isotope (although vanadium has 0.250% of a weakly radioactive isotope ^{50}V, $t_{1/2} > 10^{17}$ years and tantalum has 0.012% of isotope ^{180}Ta, $t_{1/2} > 10^{15}$ years). The third consequence of this is that the atomic weights of the three elements can be determined with great accuracy by mass spectrometry (i.e. to better than 1 part in 1/2 million, 4.6 million and 1.8 million, respectively, as seen in Table 1) [3]. Another notable feature illustrated in Table 1 is the great similarity in atomic size of the heaviest two members of the group, as a result of the lanthanide contraction. It follows that, because the atomic weight of tantalum is twice that of niobium, its density is also twice as great.

Table 1
Some properties of Group 5 elements

Property	V	Nb	Ta
Atomic number	23	41	73
Number of naturally occurring isotopes	2	1	2
Atomic weight	50.9415(1)	92.90638(2)	180.9479(1)
Electronic configuration	$[Ar]3d^34s^2$	$[Kr]4d^35s^2$	$[Xe]4f^{14}5d^36s^2$
Electronegativity	1.6	1.6	1.5
Metal radius (12-coordinate) (pm)	134	146	146
Ionic radius (6-coordinate) (pm), V	54	64	64
Ionic radius (6-coordinate) (pm), IV	58	68	68
Ionic radius (6-coordinate) (pm), III	64	72	72
Ionic radius (6-coordinate) (pm), II	79	–	–
Melting point (°C)	1915	2468	2980
Boiling point (°C)	3350	4758	5534
ΔH_{fus} (kJ mol^{-1})	17.5	26.8	24.7
ΔH_{vap} (kJ mol^{-1})	459.7	680.2	758.2
ΔH_f (monoatomic gas) (kJ mol^{-1})	510 (±29)	724	782 (±6)
Density (20 °C) (g cm^{-3})	6.11	8.57	16.65
Electrical resistivity (20 °C) (μΩ cm)	∼25	∼12.5	(12.4)

The steady increase in the number of d electrons available for metal–metal bonding in the bulk metals leads, in each case, to a higher melting point, boiling point and enthalpy of atomisation when compared to the preceding elements in each row. Indeed, these quantities reach their maximum in this and the following group.

5. Chemical reactivity and trends

The elements of Group 5 are in many ways similar to their predecessors in Group 4. They react (albeit at high temperatures) with most non-metal elements giving products which are frequently interstitial and non-stoichiometric. Their general resistance to corrosion is largely due to the formation of surface films of oxides that are particularly effective in the case of tantalum. Unless heated, tantalum is appreciably attacked only by oleum, hydrofluoric acid or, more particularly, by a mixture of hydrofluoric and nitric acids. Fused alkalis will also attack it. Vanadium and niobium are attacked by most hot concentrated mineral acids but are resistant to fused alkali.

The most obvious factor in comparing the chemistry of the three elements is again the very close similarity of the second and third members (Nb and Ta) although slight differences can be discerned. The stability of the lower oxidation states decreases as the group is descended. As a result, although all three elements show formal oxidation states all the way from +5 down to −3, the most stable one for vanadium under normal conditions is the +4; even the +3 and +2 oxidation states have well a characterised cationic aqueous chemistry, although such species are strongly reducing. By contrast, most of the chemistry of niobium and tantalum is confined to the group oxidation state of +5. Of the halogens, only the strongly oxidising fluorine produces a pentahalide of vanadium, and the other vanadium (V) compounds are based on the oxohalides and the pentoxide. The pentoxide also gives rise to the complicated but characteristic aqueous chemistry of the polymerised vanadates (isopolyvanadates) which anticipates the even more extensive chemistry of the polymolybdates and polytungstates in the next group; this chemistry is only incompletely mirrored by niobium and tantalum. I will not discuss this burgeoning field further.

The +4 oxidation state, which for niobium and tantalum is best represented by their halides, is most notable for the uniquely stable vanadyl ion, VO^{2+}, which retains its identity throughout a wide variety of reactions and forms many complexes. Indeed it is probably the most stable diatomic ion known in the whole of chemistry. Likewise, the mononuclear cationic and anionic complexes of vanadium in the +3 and +2 oxidation states find no counterpart in niobium and tantalum. Instead, these latter elements tend to form series of cluster compounds based on octahedral M_6X_{12} units. This is largely a consequence of the great strength of metal–metal bonding in this part of the periodic table (see Table 1). Similar cluster compounds are also found for the neighbouring elements molybdenum and tungsten.

Compounds containing metal–carbon σ-bonds are frequently unstable and do not give rise to an extensive chemistry in this group. Nevertheless, reduction of MCl_5 or MCl_3 under an atmosphere of CO yields salts of the octahedral anions $[M(CO)_6]^-$ which have the noble gas electronic configuration. In the case of the vanadium salt (but not those of Nb and Ta), acidification and extraction with petroleum ether yields volatile, blue-green, pyrophoric crystals of the neutral carbonyl $V(CO)_6$. Unlike other formally odd-electron transition metal carbonyls, this does not attain the noble gas configuration by dimerisation and formation of a M–M bond: it is, in fact, unique among monomeric carbonyls in being paramagnetic.

With the cyclopentadienyl ligand, vanadium forms the simple sandwich compound "vanadocene", $[V(\eta^5\text{–}C_5H_5)_2]$. This is dark violet, paramagnetic (three unpaired electrons) and extremely air-sensitive. The cyclopentadienyl chemistry of niobium and tantalum is more complicated and polynuclear forms often prevail [3].

Some of the most fascinating and important chemistry of this group involves the extensive formation of non-stoichiometric oxide phases and solid-state catalytic systems. This is, of course, the major theme of this conference so it would be inappropriate of me to attempt to summarise the many exciting and technologically significant studies that will be discussed during the next three days. All of you are more knowledgeable about these aspects of Group 5 chemistry than I am and I am keenly looking forward to learning of the most recent advances.

6. Biochemical aspects [9–11]

Not unrelated to the catalytic aspects of the chemistry of Group 5 is the biochemical activity of one of these elements. Two classes of vanadium enzymes have been found in nature: vanadium nitrogenases and vanadate-dependent haloperoxidases. In the former, vanadium is in a medium to low oxidation state as an integral part of a (Fe, V, S) cluster with the vanadium in an essentially octahedral coordination. In the latter, vanadium is in the +5 oxidation state in an environment dominated by oxygen functions and its coordination geometry changes between trigonal–bipyramidal and tetragonal–pyramidal. Thus, vanadium is an essential element for certain species, including humans, though there is less than 0.1 mg of it in the normal adult human. The average daily intake of vanadium in our food is about 40 μg, which is more than enough for the body's requirements which is probably as little as 2 μg per day. Vanadium does not accumulate in any particular organ; its role is to act as a regulator of one of the enzymes that govern the way sodium operates in the body. It has also become apparent during the past 25 years that vanadium acts as an enzyme inhibitor of ATPases, probably by substituting vanadate for phosphate, though it is not yet completely clear why vanadium is essential for humans.

Marine worms absorb vanadium and one tunicate species, *Ascidia nigra*, has blood cells with 1.5% vanadium. It has the ability to concentrate vanadium to 10,000 times higher than in the surrounding seawater. Even more remarkably, vanadium makes up 10% of the blood cell pigment of the sea cucumber, *Holothroidea*. In the plant world vanadium plays a vital role in the fixation of dinitrogen; it occurs widely in azotobacteria as part of the (Fe, V, S) cluster which acts as an alternative to molybdenum in the cofactor of some nitrogenases.

Neither niobium nor tantalum has any known biological role, though we take in enough of them to be measurable in our body: about 1.5 mg of niobium and 0.2 mg of tantalum in total. They also seem to be accumulated by certain tunicates (ascideans). Tantalum metal is well tolerated by the body and consequently has been used in many ways during surgery, for example as plates for the repair of skull fractures, as bolts to fasten broken bones, and as wire for sutures for torn ligaments.

7. The chemistry of dubnium

Finally, I want to mention briefly the emerging chemistry of the heaviest element of Group 5. It is worth noting that, because of the numerology of the periodic table, a helpful mnemonic for the trans-lawrencium elements 104–112 is that their group number is 100 less than their atomic number. Thus dubnium, element 105 is in Group 5. The first experiments on the chemistry of dubnium were carried out in the early 1970s by Ivo Zvára's group in Dubna using the isotopes ^{260}Db ($t_{1/2} = 1.5$ s) and ^{261}Db ($t_{1/2} = 1.8$ s). They combined cyclotron physics with the novel technique of thermochromatography (a thermal gradient variant of gas-phase chromatography). The recoiling product atoms were continuously chlorinated in situ with gaseous $NbCl_5$ or $ZrCl_4$ within a fraction of a second from the instant of formation of the dubnium atom. The formed chloride molecules were then swept outside the cyclotron by a stream of nitrogen gas, through a narrow, heated, 4 m long tube and then through a filter to a flat chamber provided on both sides with mica spontaneous-fission detectors. The observed volatility of the product chlorides resembled that of $NbCl_5$ rather than $HfCl_4$ in the preceding group, suggesting that element 105 was, indeed, homologous with niobium and tantalum.

No further work on the chemistry of dubnium was reported until 1988 when the first studies of its aqueous solution chemistry were published [12]. Using the 34 s isotope formed by the cyclotron reaction ^{249}Bk(^{18}O, 5n)^{262}Db, some 800 manual experiments (taking about 50 s each) were performed. It was found that, after fuming with concentrated nitric acid, atoms of element 105 sorbed on glass surfaces just like niobium and tantalum but unlike zirconium and hafnium in the preceding group. Extraction studies also confirmed its affinity to Group 5, though the element appeared closer to niobium than to tantalum, perhaps due to the influence of relativistic effects. Later work using computer controlled procedures reduced the timescale to <40 s per experiment [13]: halide complexation and extraction behaviour of dubnium appeared to be most like protactinium, a pseudo Group 5 element [14]. The elegant design and technical virtuosity of these manual and subsequent computer-controlled experiments are quite remarkable.

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