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The group VIII platinum-group metals and the Periodic Table

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Abstract The six platinum group metals (pgms: ruthenium, rhodium, palladium, osmium, iridium and platinum) posed a number of problems for 19th-century chemists, including Mendeleev, for their Periodic classification. This account discusses the discovery of the pgms, the determination of their atomic weights and their classification.

Keywords Periodic Table · Platinum · Ruthenium · Rhodium · Palladium · Osmium · Iridium · Mendeleev

Introduction

Of the six platinum group metals (often called pgms by chemists or PGEs—platinum group elements—by geologists), platinum is the only one to date from antiquity. The other five are unusual in that they were all isolated by their discoverers as the elements (in addition to some compounds), so that one of the great controversies in attributing the true "discovery" of an element—whether the recognition of a compound that is unlike any other known at the time suffices, or the isolation of the element is a necessity—does not arise for them.

In a belated commemoration of the centenary of Mendeleev's death, the author published in 2008 a paper which discussed how Mendeleev and some of his predecessors had accommodated the pgms in their Periodic systems, and commented on the chemistry of their man-made *trans*-uranic congeners, ^{108}hassium, ^{109}meitnerium and ^{110}darmstadtium (Griffith 2008). Here the congeners are omitted, but the arrangement of the pmgs within the Periodic Table and the features which posed a challenge to its designers are discussed in more depth.

The periodic sequence of the pgms is used throughout, *viz.* ruthenium—rhodium—palladium, osmium—iridium—platinum.

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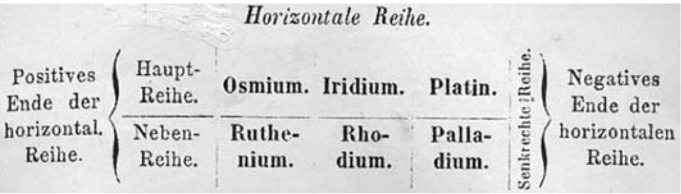
Discovery of the pmgs and determination of their atomic weights

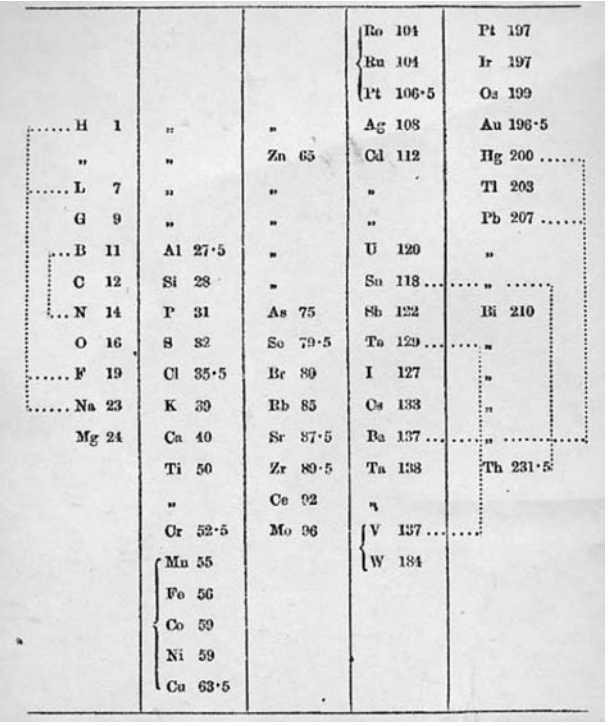
Before any sensible classification of elements could be made an accurate assessment of their atomic weights was necessary: atomic weights are useful though not a *sine qua non* for this purpose, but the more crucial atomic number was of course not known to 19th century chemists. There is a useful historical survey of atomic weights for all the elements known before 1897 (Clarke 1897). The values used by Odling (Odling 1864) and Mendeleev (Mendelejeff 1871) in their tables are shown below in Figs. 1 and 2. The current recommended values are published by the International Union of Pure and Applied Chemistry (IUPAC 2007a).



Horizontale Reihe.

Positives Ende der horizontal. Reihe.	Haupt-Reihe.	Osmium.	Iridium.	Platin.	Senkrechte Reihe.	Negatives Ende der horizontalen Reihe.
	Neben-Reihe.	Ruthenium.	Rhodium.	Palladium.		

Fig. 1 Claus's arrangement of the platinum group metals of 1860 (Claus 1860b)



			Ro 104	Pt 197
			Ru 104	Ir 197
			Pt 106·5	Os 199
H 1	"	"	Ag 108	Au 196·5
"	"	Zn 65	Cd 112	Hg 200
L 7	"	"	"	Tl 203
G 9	"	"	"	Pb 207
B 11	Al 27·5	"	U 120	"
C 12	Si 28	"	Sn 118	"
N 14	P 31	As 75	Sb 122	Bi 210
O 16	S 32	Se 79·5	Te 129	"
F 19	Cl 35·5	Br 80	I 127	"
Na 23	K 39	Rb 85	Cs 133	"
Mg 24	Ca 40	Sr 87·5	Ba 137	"
	Ti 50	Zr 89·5	Ta 138	Th 231·5
	"	Ce 92	"	
	Cr 52·5	Mo 96	V 137	
	Mn 55		W 184	
	Fe 56			
	Co 59			
	Ni 59			
	Cu 63·5			

Fig. 2 William Odling's table of elements from 1864 (Odling 1864)

Ruthenium

This was the last of the pgms to be discovered, although several candidates for it had been announced earlier (Griffith 1968; Hödrejärv 2004; Pychkov 1996). It was finally isolated by Carl Carlovich Claus (1796–1864, then chemistry professor at the University of Kazan, now in Tatarstan), who fused with KNO_3 the *aqua regia*-insoluble residue of concentrated platinum ore (“platina”) giving a solution of $K_2[RuO_4]$ from which oxides, chlorides, etc. were isolated. He made the metal by reduction of RuO_2 in H_2, and named it Ruthenium in honour of his native land (*Ruthenia*, the Latin for Russia) (Claus 1845a). He assigned it an atomic weight of 102.4 based on the $Na_3[RuCl_6]/H_2$ reaction (Claus 1845b), later raising this to 104 (Claus 1860a); modern value 101.07(2) (IUPAC 2007a).

Palladium

This was discovered by William Hyde Wollaston FRS (1766–1826) in 1802, though he did not publish the discovery until 1805 (Wollaston 1805). The story of its discovery is a highly unusual one possibly involving some chicanery and has often been told; the author has summarised the main points (Griffith 2003). Wollaston isolated it from a solution of platina in *aqua regia,* removed platinum by addition of ammonium chloride to precipitate $(NH_4)_2[PtCl_6]$, then added $Hg(CN)_2$ to the filtrate. The precipitated $Pd(CN)_2$ was ignited to give the metal. He named it after a recently discovered asteroid, Pallas.

Berzelius’ first atomic weight determination was in 1813; another one followed in 1826 (Berzelius 1826) but his improved value of 106.7 was from reduction of $K_2[PdCl_4]$ by H_2 (Berzelius 1828); modern value 106.42(1) (IUPAC 2007a).

Rhodium

This too was discovered and isolated by Wollaston (Wollaston 1804); see (Griffith 2003) who dissolved platina in *aqua regia*, added NH_4Cl to remove Pt as $(NH_4)_2[PtCl_6]$; addition of zinc to the filtrate gave a precipitate of base metals and other pgms. Dissolution of this in more *aqua regia*, addition of NaCl and evaporation of the filtrate gave the rose-red $Na_3[RhCl_6]$; addition of zinc to this gave the metal. It was named after the Greek *ρόδου*, *rhodos*, a rose, from the colour of $RhCl_3 \cdot 3H_2O$.

Berzelius first determined the atomic weight of rhodium in 1814, again in 1818 and 1826 (Berzelius 1826), but his most accurate value of 104.4 came from the $K_2[RhCl_5(H_2O)]/H_2$ reduction (Berzelius 1828); modern value 102.9055(2) (IUPAC 2007a).

Osmium and iridium (Tennant 1804)

Smithson Tennant (1761–1815) (Usselman 2005; Griffith 2004) showed that the black material remaining after dissolution of platina in *aqua regia* succumbed to molten NaOH at red heat. Aqueous leaching gave a black precipitate and a yellow solution. The precipitate was treated with HCl and the residue again fused with NaOH and extracted with aqueous NaCl, giving dark red crystals of $Na_2[IrCl_6] \cdot nH_2O$ which he reduced to the metal. This he called Iridium after the goddess Iris who displayed in her eyes the colours of the rainbow (the extraction of Ir involved many colour changes). The yellow solution from the first NaOH fusion contained $Na_2[OsO_4(OH)_2]$ and acidification of this gave the very volatile, pungent-smelling OsO_4 which he reduced to the metal. He called it Osmium from the Greek *οσμη*—*osme*, a smell.

Their atomic weights were first determined from the $K_2[OsCl_6]/H_2$ and $K_2[IrCl_6]/H_2$ reactions as 196.2 (osmium) and 197.6 (iridium) and (Berzelius 1828). The modern IUPAC values are 190.23(3) and 192.217(3) respectively (IUPAC 2007a).

Platinum

This is a genuine "metal of antiquity", known by the pre-Columbian inhabitants of what is now Ecuador and Colombia who mined it together with silver—indeed its original name *platina* is derived from a diminutive of the Spanish *plata* (silver). It was first recognised in Europe as a new material by the Italian physician Julius Caesar Scaliger (1485–1558) who wrote of "*a substance which it is has not hitherto been possible to melt by fire or by any of the Spanish art*s". The remarkable story of the realisation of its existence and its recognition by Europeans has been well told (McDonald and Hunt 1982).

John Dalton (1766–1864) assigned an atomic weight of 100 to Pt (using H = 1), commenting that *one would be inclined to think it must be more* (Dalton 1808). However as with the other pgms, it was Berzelius' values of 1826–1828 for $K_2[PtCl_6]/H_2$ which formed the foundation of all future determinations for the pgms and other elements. (Berzelius 1826, 1828). His preferred value was 197.6. In his influential paper of 1858 Cannizzaro gave a value of 196.7 (Cannizzarro 1858); modern value 195.084(9) (IUPAC 2007a).

Trends in atomic weights of the pgms

Johann Döbereiner (1780–1849, professor of chemistry at Jena) noted similarities in the chemical behaviour of "triads" of elements, in which the equivalent weight of the middle element lay roughly halfway between those of the other two. In 1829, when he used his equivalent weights for these metals (based on oxygen = 100) to demonstrate that the "triads" Pt–Ir–Os and Pd–Rh–"pluran" existed, he found the equivalent weight of the middle element to be roughly the average of the outer two (Döbereiner 1829; Leicester and Klickstein 1952). "Pluran" had been reported together with two other "new" elements in 1827 by Gottfried Osann (1796–1866). It may possibly have contained some ruthenium, but Berzelius was unable to confirm the novelty of these three elements, and Osann subsequently withdrew his claim (Pychkov 1996; Griffith 1968).

In 1853 John Hall Gladstone (1827–1902, Fullerian professor of chemistry at the Royal Institution), noted that the Rh–Ru–Pd triad was chemically related to that of Pt–Ir–Os, while the "atomic weights" (*sic*) of the latter triad were roughly twice those of the former (Gladstone 1853). In 1857 William Odling (1829–1921), then teaching chemistry at Guy's Hospital, London, noted the similarity of Pd, Rh and Ru, that the "atomic weight" (*sic*) of Pt (98.6) was about twice that of Pd (53.2), and that Pt, Ir and Os were chemically similar—remarking, with much truth, that *many osmic reactions are altogether special* (Odling 1857).

In 1863 the analytical chemist John Alexander Raina Newlands (1837–1898) briefly discussed, in a paper signed J.A.R.N., the two pgm triads (Newlands 1863). Apart from that late paper, the last important mention of triplets of the pgms was by Claus who had discovered ruthenium (Claus 1845a) and knew more about the pgms than anyone else at that time. In 1860 he arranged the three most abundant ones in a Principal series (*Haupt Reihe*), and beneath them placed a Secondary series (*Neben Reihe*), noting also the chemical similarities of each vertical pair (Fig. 1) (Claus 1860b).

The stage was now set for a periodic classification of the pgms and indeed all elements then known.

The development of periodic classifications

Claus's table shows the correct vertical pairs, but not in the now accepted sequence, and he did not set the pgms in the context of other elements.

In 1862 Alexandre-Emile Béguyer de Chancourtois (1820–1886, professor at the École des Mines, Paris), devised a "*vis tellurique*" (telluric screw) a helix on a vertical cylinder on which symbols of the elements were placed at heights proportional to their atomic weights. Although some pgms appeared on it (Rh and Pd on one incline and Ir and Pt on another), no clear relationships between them are obvious (de Chancourtois 1862). In 1864 Newlands proposed the first of his tables, arranging the known 59 elements in order of ascending atomic weights (Newlands 1864a, b). He then published his celebrated "law of octaves", noting that the chemical properties of some elements were repeated after each series of seven, and assigned ordinal numbers to elements in the sequence of their ascending atomic weights:—an early form of the atomic number (e.g. H = 1, Li = 2 etc.) (Newlands 1865). However, although the pgms featured in Newlands's tables they were often out of place.

William Odling (born, like Newlands, in Southwark, London), whose pgm triads we have noted above (Odling 1857) drew up in 1864 a table of 57 elements with the six pgms grouped next to each other (Ro is rhodium), and was the first to arrange them in a reasonably logical way in a Periodic Table (Fig. 2) (Odling 1864).

In 1869 Julius Lothar Meyer (1830–1895, chemistry professor at Tübingen) arranged the 56 known elements in an unpublished table of 1868 with Ru and Pt, Rh and Ir, Pd and Os side-by-side. His slightly later table, published in 1870 (Meyer 1870) placed the pgms correctly despite his use of low values for the atomic weights of iridium and platinum:

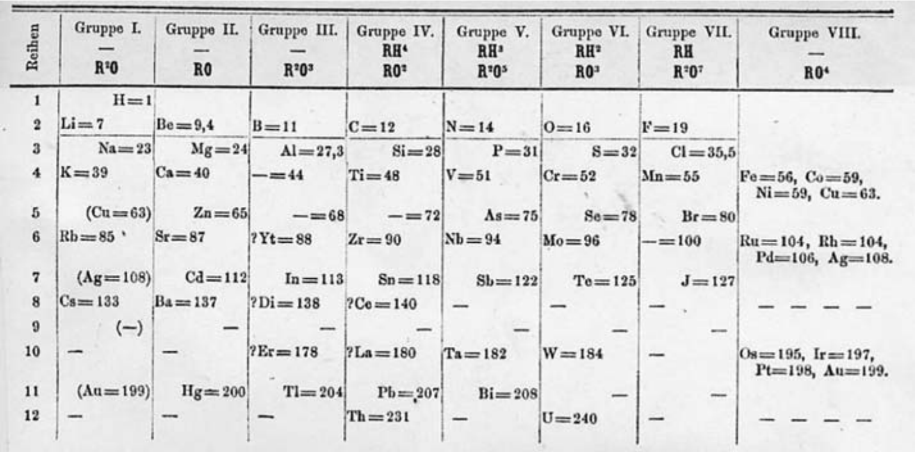
Mn = 54.8	Ru = 103.5	Os = 198.6?
Fe = 55.9	Rh = 104.1	Ir = 196.7
Co = Ni = 58.6	Pd = 106.2	Pt = 196.7

but a number of other elements lay in a sequence different from later tables.

In March 1869 Dmitri Mendeleev (1834–1907, professor of chemistry at St. Petersburg) produced his first table (here the more accessible German references are given rather than the Russian originals; the latter have been listed (Scerri 2007; van Spronsen 1969). Spellings of his name vary and are given here as in the cited references. His interest in periodicity probably dated from the Karlsruhe Congress and was cemented by writing his textbook on inorganic chemistry, part of which he finished in 1868. More than any of his predecessors in the area, he had a remarkable knowledge of the chemistry of the elements. His first published version placed the pgms together but with unusual pairings, probably because of what he perceived to be the closeness of their atomic weights (Mendelejeff 1869):

Rh 104.4	Pt 197.4
Ru 104.4	Ir 198
Pd 106.6	Os 199

The version normally regarded as his definitive table appeared in 1871, first printed in a Russian journal and then reprinted in *Annalen der Chemie und Pharmacie* in the same year (Fig. 3) (Mendelejeff 1871).



Reihen	Gruppe I. — R^2O	Gruppe II. — RO	Gruppe III. — R^2O^3	Gruppe IV. RH^4 RO^2	Gruppe V. RH^3 R^2O^5	Gruppe VI. RH^2 RO^3	Gruppe VII. RH R^2O^7	Gruppe VIII. — RO^4
1	H=1							
2	Li=7	Be=9,4	B=11	C=12	N=14	O=16	F=19	
3	Na=23	Mg=24	Al=27,3	Si=28	P=31	S=32	Cl=35,5	
4	K=39	Ca=40	—=44	Ti=48	V=51	Cr=52	Mn=55	Fe=56, Co=59, Ni=59, Cu=63.
5	(Cu=63)	Zn=65	—=68	—=72	As=75	Se=78	Br=80	
6	Rb=85	Sr=87	?Yt=88	Zr=90	Nb=94	Mo=96	—=100	Ru=104, Rh=104, Pd=106, Ag=108.
7	(Ag=108)	Cd=112	In=113	Sn=118	Sb=122	Te=125	J=127	
8	Cs=133	Ba=137	?Di=138	?Ce=140	—	—	—	— — — —
9	(—)	—	—	—	—	—	—	
10	—	—	?Er=178	?La=180	Ta=182	W=184	—	Os=195, Ir=197, Pt=198, Au=199.
11	(Au=199)	Hg=200	Tl=204	Pb=207	Bi=208	—	—	
12	—	—	—	Th=231	—	U=240	—	— — — —

Fig. 3 Mendeleev's Periodic Table of 1871 (Mendelejeff 1871)

By then Mendeleev had seen Meyer's paper and almost certainly knew of Newlands's and Odling's work, but his table represents a major advance in classification of the elements, for the first time placing the pgms in their modern sequence and in context. The dashes under the Ru–Rh–Pd–Ag listing under Group VIII misled some later workers to think that missing elements were depicted (Griffith 1968). Acceptance of his table by chemists was much helped by his astonishingly accurate predictions of the properties of the then unknown scandium (shown in Fig. 3 as "— = 44" gallium "— = 68" and germanium "— = 72"); subsequent discovery of other elements including francium, radium, technetium, rhenium and polonium owes much to his predictions. Other factors such as the successful accommodation or placement of the elements were also important, a topic recently discussed (Scerri 2007).

It is apparent from Mendeleev's table that for him (and others) the pgms, some of the transition metals and of course those of the lanthanides and actinides then known posed a problem; here we concentrate on the pgms. It is said (Pychkov 1996) that he accommodated them in his Table on the basis of Claus' arrangement of the six (Fig. 1; Claus 1860a).

He noted their very similar properties and that there were only small differences between the atomic weights of Ru–Rh–Pd and between those of Os–Ir–Pt (McDonald and Hunt 1982; Mendelejeff 1869, 1871), although modern values (IUPAC 2007a) show that these are not as close together as Mendeleev and some of his predecessors thought. Difficulties with all his Group VIII elements continued to trouble him: in 1879 he published two papers in *Chemical News* which tried to address these. In the first he split Group I–VII into left-hand "even elements" and right-hand "odd elements" blocks, with Group VIII in the centre, Cu, Ag and Au being accommodated in both VIII and in Group I of the right-hand "odd" I–VII block (Mendeleef 1879a). In the second paper he emphasised that ruthenium and osmium demonstrated octavalency in Group VIII ("RO^4"), and wondered whether FeO_4 might exist—a matter of controversy to this day (Mendeleef 1879b). He placed iron, cobalt and nickel, and the coinage metals copper, silver and gold in Group VIII, as he did in his 1871 Table (Fig. 3). In the second paper he discusses the pgms in more detail and ruefully refers to Group VIII as "special" and "independent" (Mendeleef 1879b). In his book published in 1905 essentially the same arrangement of the pgms is maintained as in Fig. 3 although it is more up-to-date with respect to elements discovered between 1871 and 1905 and includes the then recently discovered noble gases. The only difference though for the pgms is that they are given more accurate atomic weights. Copper, silver and gold are placed in parentheses under Group VIII (An is a printing error for Au) and also accommodated elsewhere. Again the three dashes appear between the two triplets (Mendeleev 1931; Laing 2008):

Ru 101.7	Rh 103.0	Pd 106.5	(Ag)
–	–	–	
Os 191	Ir 193	Pt 194.8	(An)

Some post-Mendeleev chemists were worried too by the pgms. For example, Reynolds (1844–1920) arranged the elements in a zig-zag or spiral form and envisaged them as being analogous in some ways to a vibrating system, suggesting that some triplets, e.g.

Fe	{Rh	{Ir
{Ni	{Ru	{Pt
{Co	Pd	Os

were so closely related as to be “interperiodic” (Reynolds 1886). This was picked up by Sir William Crookes (1832–1919) who, in a long address to the British Association, produced a new design, based on that of Reynolds, asserting that the same triplets Fe–Ni–Cu, Rh–Ru–Pd and Ir–Os–Pt could be regarded as “modifications of one single form of matter”. He implied that the Darwin-Wallace-Lamarckian view of Evolution could apply to elements, all evolved from a primordial form of matter (Crookes 1886). Problems with the pgms continued into the 20th century, e.g. a brief note of 1907 suggested that manganese should be placed in Group VIII (Reynolds 1907). Some other post-1900 tables are covered up to 1969 (Scerri 2007; van Spronsen 1969).

Postscript

With our understanding of atomic number and electronic orbitals the pmgs no longer seem anomalous in the Periodic Table. In vertical pairs they are indeed similar (Ru–Os, Rh–Ir, Pd–Pt), but so are other second and third-row transition metal pairs, a consequence of the lanthanide contraction (also of course unknown to Mendeleev and his contemporaries). Most second and third row transition metals are refractory and high-melting, as are the pmgs. The horizontal similarities (Ru–Rh–Pd, Os–Ir–Pt) which seemed so important in the 1860–1870 period are less apparent to us now. Thus, ruthenium and osmium exhibit no less than *eleven* oxidation states, from M^{VIII} to M^{-II}, corresponding to d electron occupancies of d^0 to d^{10} inclusive, deriving from their central position in the transition metal block: in this respect they have much more similarities to technetium and rhenium than to rhodium and iridium. The latter pair have eight oxidation states (from M^{VI} to M^{-II}, d^2 to d^{10}), while palladium and platinum have six (M^{VI} to M^0, d^4 to d^{10})., so that these three pairs are really quite distinct chemically. The important homogeneous catalytic activity of all the pgms arises mainly from their access to oxidation state and stereochemical changes, and this may in part account for their success in heterogeneous catalysis. Of course they do stand as an important and interesting group, but we no longer perceive them as chemically anomalous.

For many years “modern” tables showed Group VIII as containing the nine elements Fe, Co Ni and the pgms, with Cu, Ag, and Au designated as Group IB. The modern and now generally accepted version of the Table (Connelly et al. 2005; IUPAC 2007b) was proposed in 1988 (Fluck 1988) and recommended by IUPAC in 1990 (Leigh 1990);

interesting reflections on IUPAC and the Table are about to be published (Leigh 2009). In this modern form Mendeleev's Group VIII gives way to Groups 8, 9 and 10 with the pgms occupying Groups 8 (Ru–Os), 9 (Rh–Ir) and 10 (Pd–Pt) (IUPAC 2007b). Thus in 1988, the celebrated inorganic textbook "Advanced Inorganic Chemistry" (Cotton and Wilkinson 1988) used the earlier system but the sixth edition (Cotton et al. 1999) uses the form in which the pgm vertical pairs are in Groups 8, 9 and 10, as in (IUPAC 2007b).

Mendeleev's iconic table of 1871 (Fig. 3; Mendelejeff 1871) is the definitive Periodic Table of the 19th century and the basis of all later ones, and it remains recognisable as the kernel of today's tables. It comfortably and correctly accommodates the pgms.

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