# The contributions to original research in chemistry by Professor Sir Geoffrey Wilkinson FRS 1921–1996

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Professor Sir Geoffrey Wilkinson was one of if not the most influential of inorganic chemists in the post-war era. This article serves to highlight his contributions in original research and does not address his influence through his famous text books or the impact arising from the exceptionally large number of graduate students and co-workers—the children, grandchildren and great grandchildren *etc.* who have been inspired by working with him during the forty-five years that he held academic posts first at MIT and Harvard and subsequently, for 41 years as the Sir Edward Frankland Professor of Inorganic Chemistry at Imperial College.

It is not intended that this article will embrace a full and detailed evaluation of the contributions encompassed in the list of some 560 publications given at the end of this article. We have set out to identify and illustrate key areas and, from time to time, to embellish with stories and observations we feel are illustrative of Wilkinson's style as a research mentor and his attitude to research. We have adopted an essentially chronological order but within this there is some overlap of themes. The headings of the sections below are:

## I. Early Life and Education

Wilkinson was always very proud of the fact that he was a Yorkshireman, and that all his forebears were Yorkshire people. His grandfather (also Geoffrey Wilkinson) came to Todmorden from Boroughbridge; his father Harry fought in the First World War, and was lucky to survive, having been left for dead in France at the battle of Bullecourt in 1917. Harry married a weaver, Ruth Crowther in 1920; members of the Crowther family had been weavers in the local mills for several generations. Geoffrey was born on July 14, 1921, the first of three children, in the village of Springside on the outskirts of Todmorden—sadly there is little left of Springside now. The family moved to the centre of Todmorden in 1926, to no. 4 Wellington Road (the house is now marked by a blue plaque erected in 1990). Todmorden is a small industrial town at the junction of three deep valleys in the centre of the Pennines, the hills and moors rising 1000 feet above the town. The population today is about 13 000, about half of that which existed before the decline of the cotton industry. His brother John speaks of the strong local pride and sense of community which Todmorden had and still has, and Wilkinson remained very fond of Todmorden all his life. His eyes would light up when he spoke of it or remembered the superb countryside such as Hardcastle Crags close by. He often returned there and had friends in the local community: he always enjoyed walking on the moors near Todmorden and fell-walking in the Lake District with family and friends.

His interest in chemistry came early in life, before he went to secondary school. He recalled being fascinated at the age of six or seven by watching his father—a house painter and decorator—mixing his painting materials (his uncle John Willy Wilkinson was also a painter, but had died at the age of 22 from accidental arsenic poisoning caused by a green copper–arsenical pigment, Paris Green, fashionable at that time). An uncle on his mother's side managed a factory making Epsom and Glauber's salts in Todmorden; Wilkinson recalled how he loved to go on a Saturday morning to tinker in the small laboratory at the factory in Todmorden—indeed the family hoped that he would eventually manage the factory, but his career was to be very different. His parents, like most people at that time, had left full-time education at the age of 12; they were determined that their children should have a better education, and Wilkinson never forgot this. He went to Roomfield School and then, having won a West Riding County Minor Scholarship in 1931, went to Todmorden Secondary School, later named Todmorden Grammar School and later still Todmorden High School. He made the most of his education there. The school had several other pupils who were later to become famous, including Sir John Cockcroft, who worked with Rutherford at Cambridge and was to become the first of the school's two Nobel Laureates-to-be in 1951. Many of the school's pupils were groomed for entrance to Cambridge, but Wilkinson made exceptional progress and was entered for a Royal Scholarship at the Imperial College of Science and Technology, London University, because the entry scholarships there were held at an earlier date than for other universities. He won the scholarship and, after a two day practical analytical chemistry examination in London, joined the college in 1939.

## II. University and Post-doctoral Years

### II.1. B.Sc. and Ph.D. at Imperial College

At Imperial Wilkinson found the competition to do well even fiercer than it had been at his school. In his year was Ralph Raphael, later to be professor of Organic Chemistry at Glasgow, and in the year before this was another Nobel Laureate-to-be, Derek Barton. His main subject was chemistry but he also studied geology as an ancillary subject, and did so well at the latter subject that he won the prestigious Murchison prize in Geology. This honour was one of which he was extremely proud of throughout his life—though he affected not to care about prizes (he received many honours and awards in his life) he would proudly show visitors this impressive bronze medal. Indeed in those early days he almost gave up chemistry in favour of geology.

He graduated in 1941 with a first class honours B.Sc., the top student of his year, and went on to do a Ph.D. under H. V. A. Briscoe (at that time the only Professor of Inorganic Chemistry in the country) on ‘Some physico-chemical observations on hydrolysis in the homogeneous vapour phase’. This rather Delphic title conceals the fact that the main substrate studied was phosgene (he remarked that Briscoe ‘directed his Ph.D. research from a safe distance’); he found that phosgene did not hydrolyse under the conditions used (25–200 °C).

### II.2. The post-doctoral years: the Atomic Energy project in Canada

Towards the end of 1941 he was selected by F. A. Paneth, acting on behalf of the Joint Recruiting Board, to join the British contingent as a scientific officer at the Atomic Energy project in Canada; he sailed out on *H.M.S. Andes* from Greenock to Halifax, Nova Scotia on 11 January 1942 . He worked first at the University of Montreal, and then in 1944 at Chalk River, Ontario when the Atomic Energy project was moved to purpose-built premises there. In Montreal and at Chalk River he was to work with many celebrated names—Cockcroft (from his old school at Todmorden), Bertrand Goldschmidt, Charles Coryell, Alfred Maddocks (later to go to Cambridge), Jules Guéron and Auger amongst them, and two figures later found to be spies of the Soviet Union, Alan Nunn May and Bruno Pontecorvo. He also met Glenn T. Seaborg on a number of occasions. With Bill Grummitt he worked on the famous ‘double-humped’ curve in which fission product yields were plotted as a function of mass number from the slow fission of $^{235}$U, and this seminal work provided the material for one of his first papers.[1] His very first paper, on the growth of $^{140}$La from $^{140}$Ba, was on work completed in 1944 but published, when declassified, in 1947.[2]

### II.3. The Lawrence Livermore Laboratory and Massachusetts Institute of Technology

After the war Wilkinson returned briefly to Britain to take his Ph.D. examination and then went to the United States, to the Lawrence Livermore Laboratory at the University of California at Berkeley, where he worked with Glenn Seaborg. His first year at Berkeley was totally self-financed but then Seaborg (another Nobel Laureate-to-be) obtained funding for him. He worked on the production of neutron-deficient isotopes of the transition elements and the lanthanides,[1–19,22,23] and it was said (by Seaborg) that he made more artificial isotopes than anyone else. In his radiochemical references [1–3,5–10,14–19] some 89 are reported. A number of these papers are with his first student, Harry Hicks, who later joined GEC in Richland, Washington. It was from this period that Wilkinson started to amass his vast knowledge of inorganic chemistry: in those days nuclear chemists had to know in great detail the chemistry of the transition metals, the lanthanides and the actinides in order to devise appropriate procedures to separate their isotopes. A glimpse of this is provided by one of his early papers.[22] One of his nuclear transmutations was of platinum to gold,[7] which caught the public imagination after a report in 1948 in the *San Francisco Chronicle* (‘Scientist discovers gold mine in the cyclotron’).

In 1949 the first post-war conference on Nuclear and Radiation Chemistry was organised by the Royal Society in Oxford; Wilkinson gave a paper there and, as his thirtieth birthday approached, used the occasion to investigate the possibility of employment in this country. Paneth offered him a research fellowship at Durham but he declined this, and Briscoe advised him that there might not be much future in his kind of nuclear chemistry since he would always be dependent on the physicists running the cyclotron. Briscoe suggested that he concentrate on inorganic chemistry, perhaps thinking that Wilkinson might turn out to be a suitable future occupant of his own chair at Imperial. So he turned to co-ordination chemistry and contacted an old friend of his, Charles D. Coryell at MIT, who offered him a research contract for 1950–1951. His first paper there concerned the isolation of an unusual zerovalent nickel complex, $[Ni(PCl_3)_4]$,[24] made from $[Ni(CO)_4]$ and phosphorus trichloride (see below); he also made $[Ni(PBr_3)_4]$ and $[Ni(PF_3)_4]$.[20] Then, in what is probably his only foray into bioinorganic chemistry, he wrote a note on a haemoglobin–$PF_3$ complex. It is interesting that he did not use his own blood for the latter experiments but that of Charles Coryell (who is acknowledged in the paper for the donation).[25] In the summer of 1951 he married Lise Schou, a Danish plant physiologist whom he had met at Berkeley and who was to be a great support to him for the rest of his life. In September 1951 he was appointed Assistant Professor of Chemistry at Harvard, and was amused to find that the desk given to him in his office at Harvard had belonged to Theodore Richards, who had been Briscoe’s mentor and who was awarded the Nobel Prize in 1914 for his determinations of atomic weights.

It was here that he did the research which was to make him famous and eventually to lay the cornerstone of his Nobel Prize.

## III. The Birth of Metallocene Chemistry

### III.1. The Harvard years: the development of ferrocene chemistry and other η-cyclopentadienyl–transition metal compounds

The recognition by Wilkinson and Woodward of the unique sandwich structure of bis(cyclopentadienyl)iron, a compound first reported by Kealy and Pauson, and the appreciation of the further implications was probably the most crucial step in Wilkinson’s career and was the inspiration that launched the modern era of organo–transition metal chemistry. This field was to become the most rapidly developing area of chemistry for the next twenty-five years or so. The brief communication by Wilkinson, Rosenblum, Whiting and Woodward [26] clearly proposed the sandwich structure (Fig. 1) which gave the iron centre an 18-electron count. The heat of formation and redox properties [30] of ferrocene were determined.[27] Wilkinson has provided a vivid personal account of the first four months of sandwich chemistry at Harvard which, in the final paragraph, records the first paper by E. O. Fischer.[320]

In rapid succession Wilkinson described ruthenocene and ruthenocenium salts,[28] cobaltocenium salts [29] and nickelocene.[31] The bis(cyclopentadienyl) dibromides of titanium,[31] zirconium [31] and vanadium [31] were prepared from the metal tetrachlorides and cyclopentadienylmagnesium bromide. The bis(cyclopentadienyl) cations of rhodium(III) and iridium(III) were prepared from the tris(acetylacetonate) derivatives of the met-

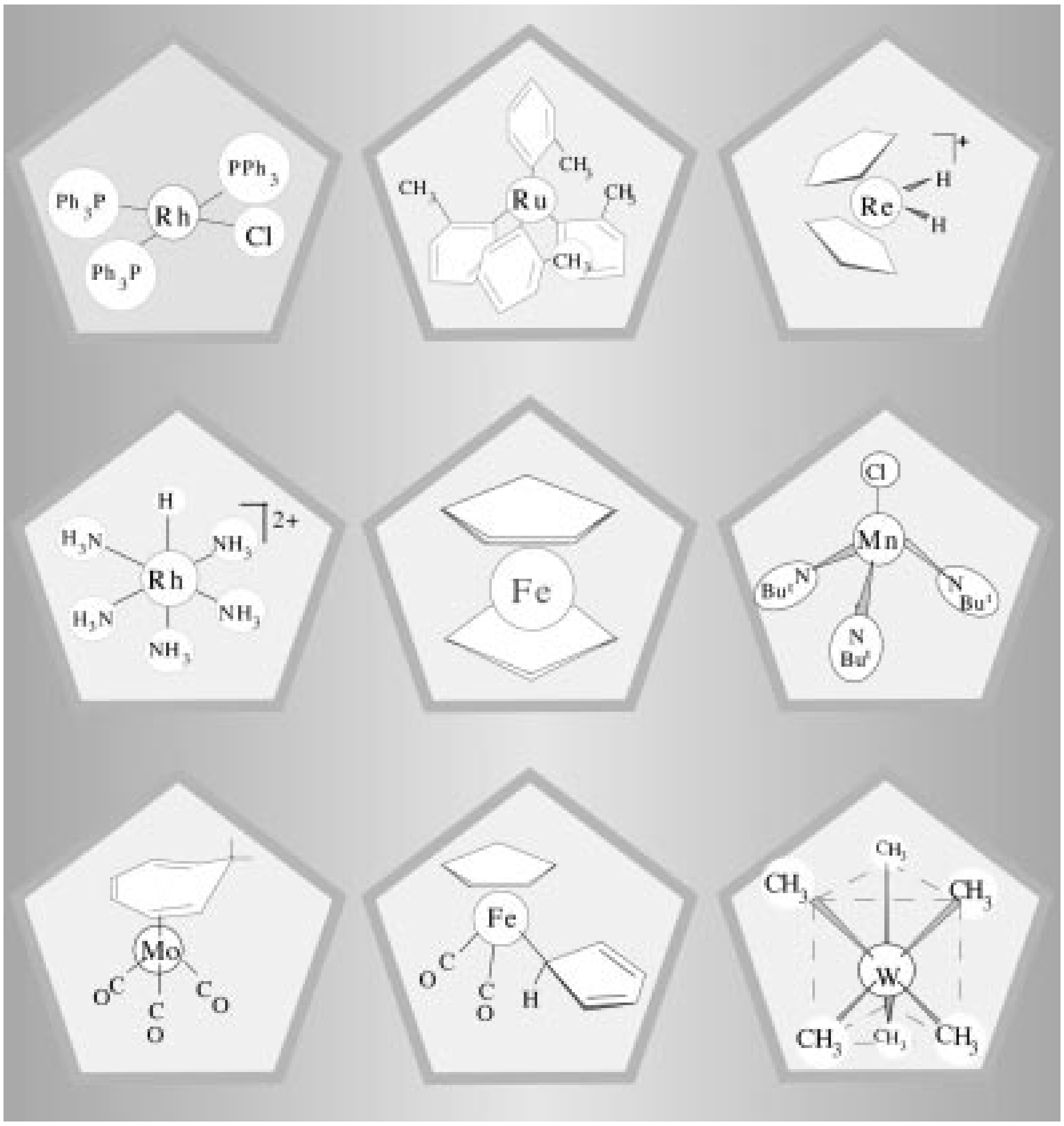


**Fig. 1** Selection of compounds prepared by Wilkinson which exemplify important areas and contributions. First row: $[Rh(PPh_3)_3Cl]$, Wilkinson's catalyst for homogeneous hydrogenation of olefins; [Ru(*o*-methylphenyl)$_4$], an example of a high oxidation state homoleptic aryl derivative; the cation $[Re(\eta\text{-}C_5H_5)_2H_2]^+$ representing the first organometallic transition-metal hydride and the first example of a metal protonation reaction, also the first compound for which the characteristic high-field shift of a hydrogen attached to a transition metal was observed in the $^1H$ NMR spectrum. Second row: the cation $[Rh(NH_3)_5H]^{2+}$, Wilkinson was the sole author of the paper describing this classic stable hydride derivative of a simple co-ordination compound; ferrocene, the first sandwich compound; the first example of a $Mn^{VII}$ compound since Glauber reported the permanganate anion. Third row: the first seven-membered metal–hydrocarbon compound; the first fluxional molecule, the 1,2 shift, 'ring-clicking' mechanism was proposed to account for the observation of a single band in the $^1H$ NMR spectrum of the $\sigma\text{-}C_5H_5$ ring; the most elegant example of a homoleptic transition-metal alkyl compound

als with cyclopentadienylmagnesium bromide and the cations were shown to be stable in hot concentrated nitric acid.[32] Chromocene and the first cyclopentadienyl derivatives of metal carbonyls, namely the cyclopentadienyl carbonyl derivatives of chromium, molybdenum and tungsten were prepared from the direct reactions between cyclopentadiene vapour and the vapours of the hexacarbonyls in a hot tube (280–350 °C).[33] The stoichiometry of the tungsten compound was determined correctly as $[W(\eta\text{-}C_5H_5)(CO)_3]_2$ but the molybdenum compound was described as having only five carbon monoxide ligands.[33] The bis(cyclopentadienyl) derivatives of molybdenum and tungsten appeared in early 1954.[37] The first full paper to describe the bis(cyclopentadienyl) halide derivatives of Ti, Zr and V[39] also reported the previously unknown cyclopentadienylniobium and -tantalum derivatives with the formulation [Nb(or Ta)$(\eta\text{-}C_5H_5)_2Br_3$].[39] An important advance was the use of potassium cyclopentadienide in tetrahydrofuran a reagent which Wilkinson came across whilst reading his favourite[320] textbook at that time, namely N. V. Sidgwick's *The Chemistry of the Elements*.

Prior to his appointment to Imperial he spent a short sabbatical leave at the University of Copenhagen in 1954 where he worked in the laboratory which had once belonged to S. M. Jörgensen, Werner's great rival at the turn of the century. The upward draught in the fume cupboard there was still accomplished by having flaming gas burners at the top, and Wilkinson with his organometallic research using flammable organic solvents had a number of fires. It was here however that he synthesised $[Re(\eta\text{-}C_5H_5)_2H]$, and on his return to Harvard he measured its proton NMR spectrum which showed, for the first time, the high-field shift due to the metal hydride.[42,79] Furthermore bis(cyclopentadienyl)rhenium hydride provided the first example of the ability of transition metals to undergo protonation at the metal to give a metal–hydrogen bond, to form the cation $[Re(\eta\text{-}C_5H_5)_2H_2]^+$ (Fig. 1). Manganocene was also described in the same year.[36]

Although not published until 1956, the experiments which established the existence of cyclopentadienyl compounds of copper and mercury,[51] the tris(cyclopentadienides) of scandium, yttrium, the rare earth elements,[43,54] and the actinides thorium and uranium,[60] in addition to the cyclopentadienyl carbonyl compounds of vanadium, manganese,[44] iron,[44]

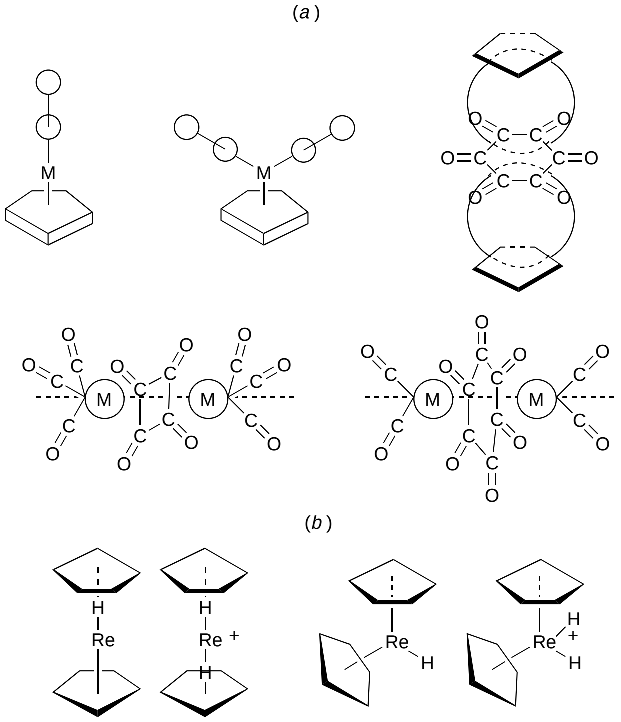


**Fig. 2** Some early structures proposed by Wilkinson: (*a*) metal carbonyl compounds and (*b*) structures considered for the compound $[Re(\eta\text{-}C_5H_5)_2H]$ and the cation $[Re(\eta\text{-}C_5H_5)_2H_2]^{+\bullet}$, the proposed structures were later confirmed

cobalt[44] and the first cyclopentadienyl nitrosyl derivatives of chromium[49] and manganese[44,49] were also carried out at Harvard. The first alkyl derivatives, for example, $[Cr(\eta\text{-}C_5H_5)(NO)_2Me]$[53] and especially the iron compounds $[Fe(\eta\text{-}C_5H_5)(CO)_2R]$,[59,61] where R = Me, Et or Ph, were prepared and were notable for their thermal stability. A particularly interesting compound was the first example of a σ-cyclopentadienyl ligand in the compound $[Fe(\eta\text{-}C_5H_5)(CO)_2(\sigma\text{-}C_5H_5)]$ (Fig. 1) which was first reported by Pauson. The proton NMR spectrum of this compound showed a sharp single band for the $\eta\text{-}C_5$ ring and also a single but broader band for the other $C_5$ ring. However, the infrared spectrum suggested the latter ring had several bands associated with C–H stretching modes. This dichotomy led Wilkinson to suggest the idea of a fluxional 'ring whizzing' process[61] and this bold hypothesis was later confirmed by variable-temperature NMR studies. Also, whilst at Harvard, the heats of combustion of ferrocene, bis(benzene)-chromium and binary metal carbonyls were reported,[27,63,72,73] the first such quantitative thermodynamic data for organo-transition-metal compounds.

The structures proposed for some of these compounds are shown in Fig. 2 where they are drawn in the manner shown in the original papers. Access to X-ray crystal-structure determination was very limited and the most available tool for providing structural evidence in metal carbonyl compounds was infrared spectroscopy—a 'sporting' method.

It was this body of work on the cyclopentadienyl–transition metal compounds of which a substantial proportion of the experimentation was carried out by Wilkinson himself which led to his appointment to the Chair in Inorganic Chemistry at Imperial College—and later to the award of the Nobel prize jointly with E. O. Fischer who had made a parallel contribution to the early development of the field and, notably, the synthesis of the compound bis(benzene)chromium.

He was appointed in 1955 to Briscoe's old chair at Imperial College—as had been the case in 1940, this was still the only established chair of inorganic chemistry in Britain—and arrived in January 1956 to take up his appointment. At 34 he was one of the youngest professors that the college has ever had. The first paper of his to bear the college address was on the infrared spectra of manganese and rhenium decacarbonyls,[55] but the work was almost certainly done at Harvard. The first paper to concern research done at Imperial is an unusual one, on the nature of the blue species formed in a number of ethers by sodium–potassium alloy.[68] This was followed by a paper on the infrared spectrum of $K[OsO_3N]$ and $K[OsO_3{}^{15}N]$, work carried out by the two authors (Wilkinson and Jack Lewis, later Sir and then Lord, Jack Lewis) themselves.[69]

After his arrival at Imperial College, Wilkinson's interests rapidly diversified into several directions and, perhaps surprisingly, he only continued work on cyclopentadienyl–transition metal chemistry for a few more years. He consolidated his interests in cyclopentadienyl–transition metal hydrides by the synthesis of the compounds $[M(\eta\text{-}C_5H_5)_2H_x]$ (where M = Re,[79] $x = 1$, Mo or W,[108] $x = 2$ and M = Ta,[130,137] $x = 3$). He explored the protonation of (cyclopentadienyl)carbonylmetal derivatives and, indeed, ferrocene itself using strong protic acids and made extensive use of NMR spectroscopy to detect the presence in solution of hydride species.[125,142] He first demonstrated the exo-addition of nucleophiles to the η-cyclopentadienyl ring in cationic transition-metal derivatives and the existence of the η-cyclopentadiene ligand in $[Co(\eta\text{-}C_5H_5)(\eta\text{-}C_5H_6)]$.[96]

Thus in the space of less than ten years Wilkinson had established the extraordinary stability of the bonding between transition metals and the η-cyclopentadienyl ligand and provided the first examples for most of the d block transition metals, the lanthanides and the actinides thorium and uranium. This was a period of exceptional productivity.

Since those days the metal chemistry of η-cyclopentadienyl metal derivatives has expanded into an extraordinary variety of exciting areas of chemistry. The seeds for the current excitement and industrial application of metallocenes in homogeneous Ziegler–Natta catalysis for olefin polymerisation were sown in these years. Another recent industrial application is the use of ferrocene derivatives as mediators in the new generation of glucose sensors. This technology takes advantage of the substitutional inertness yet redox active nature of ferrocenes.

### III.2. Other transition-metal derivatives of unsaturated hydrocarbons

An important compound which was prepared at an early stage following Wilkinson's arrival at Imperial College was the first seven-membered ring compound $[Mo(\eta\text{-}C_7H_8)(CO)_3]$.[71,85] This was the first significantly new organometallic compound to be prepared at Imperial College and substantially raised the morale of the new and inexperienced students who had laboured in vain until that time. Later azulene metal compounds were prepared.[84,115] Wilkinson also investigated unsaturated fluorocarbons as ligands to transition metals.[133,139,145,154]

## IV. Co-ordination Chemistry

Although Wilkinson is perhaps remembered as an organometallic chemist he did make a very considerable contribution to co-ordination chemistry (*i.e.* in complexes where no metal–carbon bond is present). Outstanding in this area, of course, is his work on Wilkinson's catalyst, $[RhCl(PPh_3)_3]$, and the later work on imido complexes. Wilkinson used to remark that much of his work concerned the three R's—rhodium, ruthenium and rhenium—and these are dealt with in separate sections below, followed by a consideration of some of the principal ligand groups that he used.

### IV.1. Rhodium complexes

As part of his radiochemical work at Berkeley he isolated the short-lived $^{106}Rh$ as one of the many fission products of $^{235}U$,[22] and in 1953 isolated salts of the $[Rh(\eta\text{-}C_5H_5)_2]^+$ cation.[32] However, he was very proud of work that he carried out him-

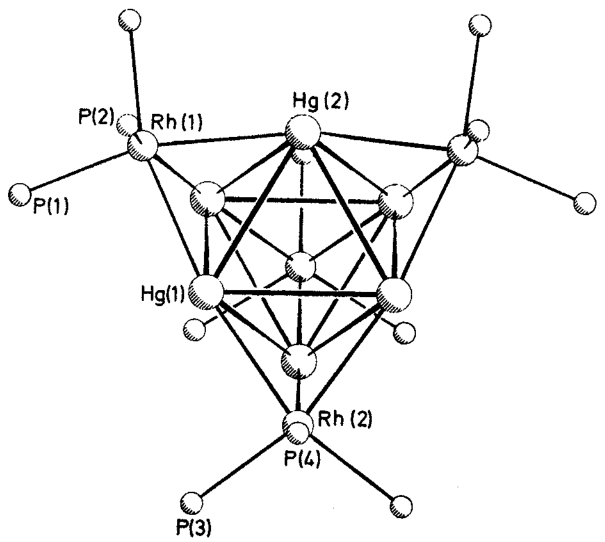


**Fig. 3** Geometry of the $[Hg_6Rh_4P_{12}]$ nucleus[386]

self in 1961 on the reaction of *cis*- and *trans*-$[RhCl_2(en)_2]^+$ (en = ethane-1,2-diamine) with sodium tetrahydroborate in aqueous solution to give $[RhHCl(en)_2]^+$ (detected by the high-field shift of the hydridic proton by $^1H$ NMR spectroscopy).[131] It was probably this work that gave rise to the (undoubtedly true) story of Wilkinson rushing into the lab, demanding a Bunsen burner and a test tube, and returning later with the tube full of a foaming brown liquid which he brandished about, crying 'Who wants a Ph.D.?'

There followed work on triethylenetetramine (trien)[151] and EDTA (ethylenediaminetetraacetate)[157] complexes of rhodium(III) and cobalt(III), and on aquation[165] of $[Rh(ox)_3]^{3-}$ (ox = oxalate). The early note referred to above[131] led to a paper of major importance in which $[RhH_2(en)_2][BPh_4]$ was isolated and the reduction of quinone to quinol by hydride transfer from $[RhH(trien)Cl]^+$ established.[155] Pyridine halogenato complexes of rhodium were made[171] and, in a joint paper with Nyholm from University College London, it was shown that some complexes previously regarded as containing rhodium(II) were in fact rhodium(III) species, *e.g.* '$[Rh(py)_6]Br_2$' is *mer*-$[Rh(py)_3Br_3]$.[187] Also dating from that period is a paper on the place of the hydride ligand in the spectrochemical series, based on the electronic spectra of a number of rhodium hydrido species,[186] polarographic work on a number of rhodium(III) complexes showing that they underwent two-electron reductions to rhodium(I) or hydridorhodium(I) complexes,[179] and circular dichroism work on the absolute configurations of cobalt(III) tris(diamine) complexes.[175] In collaboration with that wizard of platinum group chemistry, A. R. Powell of Johnson Matthey, salts of $[RhH(NH_3)_5]^{2+}$ and $[RhH(H_2O)(NH_3)_4]^{2+}$ were isolated; these these materials were used to prepare hydrido complexes of ethylenediamine and propylenediamine.[241] The presence of unidentate dithiocarbamate ligands in $[Rh(S_2CNEt_2)(PPh_3)_2]$ was postulated,[252] and $[Rh_2(OCOCH_3)_4]$ was used as a precursor for a number of other rhodium carboxylato species, *e.g.* $[Rh(OCOR)(PPh_3)_3]$ and $[Rh(OCOR)(CO)(PPh_3)_2]$.[281] The first thiocarbonyls, namely $[RhCl(CS)(PPh_3)]$,[212,231] and the first $CS_2$ complexes[218,224,231] were described. Single-crystal structures of the planar $[Rh(PMe_3)_4]Cl$ and $[RhCl(PMe_3)_3]$ were reported.[368,371] Reaction of $[Rh_2(OCOMe)_4]$ with 3,5-dimethylpyrazole (L) in acetonitrile gives $[Rh_2(L)_4]\cdot 2CH_3CN$; a structure determination was carried out and a number of adducts of the complex were prepared.[447] A remarkable cluster, $[Hg_6Rh_4(PMe_3)_{12}]$, was made by treatment of $[Rh(PMe_3)_4]Cl$ with mercury–sodium amalgam in tetrahydrofuran (THF). The crystal structure, shown in Fig. 3, revealed four opposite faces of an $Hg_6$ octahedron capped by $\{Rh(PMe_3)_3\}$ units. In the same paper the crystal structure of *trans*-$[RhCl(PPh_3)(PMe_3)_2]$ is reported.[386]

## IV.2. Catalytic hydrogenation and hydroformylation with rhodium complexes

Wilkinson's work on this topic revolutionised our view of homogeneous catalysis effected by transition-metal complexes, and constitutes some of his most celebrated work. It is well reviewed in articles by Fred Jardine (F. Jardine, *Rhodium Express*, 1997, **16**, 4; *Prog. Inorg. Chem.*, 1981, **28**, 63), and the following owes much to those reviews.

Some of the early work already referred to above forms part of this story. In 1965 he reported that catalytically small amounts of reducing agents (such as hypophosphorous acid, zinc amalgam and dihydrogen itself) would catalyse the otherwise slow substitution reactions of rhodium(III) complexes. Thus it was found possible to make *trans*-$[Rh(py)_4Cl_2]Cl$ from $RhCl_3\cdot nH_2O$ in the presence of such reductants.[189] It was shown that $RhCl_3\cdot nH_2O$ would absorb dihydrogen and convert hex-1-ene to hexane,[190] and in 1965 it was found that *fac*-$[RhCl_3(PPh_3)_3]$ would, with dihydrogen and carbon monoxide under pressure at 55 °C, convert hex-1-ene to *n*-heptaldehyde.[194] However $[RhCl_3(PPh_3)_3]$ is difficult to make, and indeed it was during an attempt to make it that $[RhCl(PPh_3)_3]$, a much more effective catalyst, was produced.[213] It was found that $[RhCl(PPh_3)_3]$ would more effectively catalyse the hydrogenation of alkenes and alkynes and also effect the hydroformylation of hex-1-yne to *n*-heptaldehyde and 2-methylhexaldehyde.[199,200] The preparation of $[RhCl(PPh_3)_3]$, later to be known as Wilkinson's catalyst, was described in a classic paper of 1966;[213] it was made from $RhCl_3\cdot nH_2O$ in ethanol with excess triphenylphosphine. This seminal paper and subsequent ones described its properties,[214,232] and the likely mechanism of catalytic hydrogenation.[213,237] Although $[RhCl(PPh_3)_3]$ is a hydrogenation catalyst (and subsequently chiral analogues of it were to be used by others for chiral hydrogenations) he was later to show that it was not a hydroformylation catalyst; the species responsible for the latter process is $[RhH(CO)(PPh_3)_3]$.[249,266,267,270] Most of the butyraldehyde [used for synthesis of bis(2-ethylhexyl)phthalate, a plasticiser for PVC] made these days uses it as the catalyst. Wilkinson did not reap the full commercial rewards of the discovery of the use of $[RhH(CO)(PPh_3)_3]$ because it can be made from almost any rhodium complex containing triphenylphosphine under the conditions of the industrial hydroformylation procedure.

## IV.3. Ruthenium chemistry

Again his first approaches to this metal were *via* radiochemistry,[1,5] followed by preparation of ruthenocene $[Ru(\eta\text{-}C_5H_5)_2]$ and the ruthenocenium $[Ru(\eta\text{-}C_5H_5)_2]^+$ cation[28] as a logical follow-up to his classic ferrocene paper.[26] Subsequently he investigated many organoruthenium complexes, and his work on the co-ordination chemistry of the element with $[SnCl_3]^-$ and carboxylates is mentioned below. A paper of great future importance was that describing the isolation of a number of ruthenium-(III) and -(II) phosphine, arsine and stibine complexes, including $[RuX_2(LPh_3)_3]$ (X = Cl or Br; L = P or Sb) and $[RuX_3(LR_3)_2(CH_3OH)]$ (L = P or As).[205] Schiff bases (L) and $[RuCl_2(PPh_3)_3]$ yield $[Ru(L)(PPh_3)_2]$ with quadridentate bifunctional bases and $[Ru(L_2)(PPh_3)_2]$ for the bidentate bifunctional bases;[342] it also reacts with a wide variety of other ligands including dithiocarbamates, amines, nitriles and carboxylates,[257] and with various nitrogen, oxygen and sulfur atom donors.[362] In later work, aluminohydride complexes $[L_3HMAlH(\mu\text{-}H)_2AlH(\mu\text{-}H)_2MHL_3]$ (M = Ru or Os; L = $PMe_3$, $PEtPh_2$ or $PPh_3$) were obtained from $[MCl_2L_3]$ and lithium aluminium hydride.[453]

Another highlight was the isolation of the first paramagnetic second-row transition-metal complexes, $[Ru_2(OCOR)_4Cl]$ (R = Me, Et or $Pr^n$).[210] A wide range of complexes was also prepared with dithiocarbamato[252] ligands. Reaction of $RuCl_3\cdot nH_2O$ with acetic acid gave $[Ru_3O(OCOCH_3)_6$-

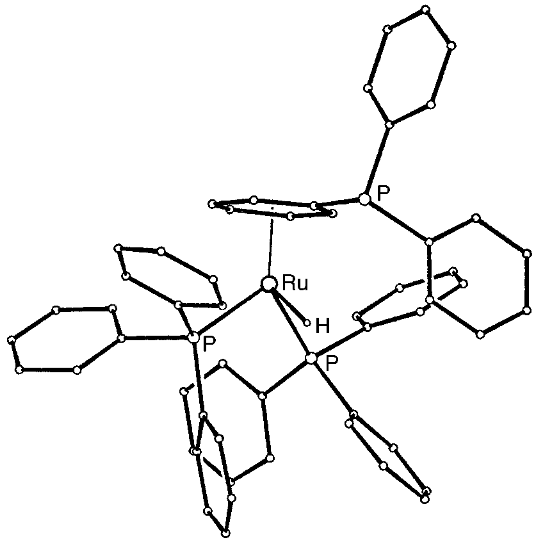


**Fig. 4** Structure of the $[RuH(PPh_3)_2(\eta\text{-}PhPPh_2)]^+$ cation[308]

$(H_2O)_3][OCOCH_3]$;[280] a polarographic study revealed a rich electrochemistry of this and related trinuclear species, mixed oxidation states ($Ru_3^{III}$; $Ru_2^{III}Ru^{II}$; $Ru^{III}Ru_2^{II}$; $Ru_3^{II}$) being involved.[296] Reactions of $[Ru_3O(OCOCH_3)_6(CH_3OH)_3]^+$ with $\pi$-acid ligands (NO, CO, $CH_3NC$ or $SO_2$) gave a variety of products,[295] while $[Ru_3O(OCOCH_3)_6(PPh_3)_3]$ in strong acids catalysed the hydrogenation of alkenes[292] in which hydridic species are involved.[317] A variety of carboxylato complexes of the form $[RuH(OCOCH_3)(PPh_3)_3]$ were found to be efficient hydrogenation catalysts for alk-1-enes.[260] In an early paper of his the crystal structure of $[RuH(PPh_3)_2(\eta\text{-}PhPPh_2)]^+$ was described,[308] as shown in Fig. 4.

The use of $[RuCl_2\{(CH_3)_2SO\}_4]$ as a precursor for a wide range of other ruthenium(II) complexes with carboxylate, bipyridyl, phenanthroline and other ligands was explored.[291] The nature of the blue solutions formed by treatment of commercial $RuCl_3{\cdot}nH_2O$ with dihydrogen was studied and it was suggested that the cluster $[Ru_5Cl_{12}]^{2-}$ gave rise to the colour.[269] A number of ruthenium carboxylates $[Ru_2(\mu\text{-}OCOR)_4L_2]$ [R = H, Me, $CH_2Cl$, Et or Ph; L = $H_2O$, $CH_3OH$, THF, $(CH_3)_2CO$ or $CH_3CN$] were made from the blue reduced solution of $RuCl_3{\cdot}nH_2O$ in methanol. The complexes are paramagnetic, and single-crystal X-ray studies were made of some of them.[450]

## IV.4. Rhenium complexes

A lifelong interest in rhenium perhaps dates from an early radiochemical paper in which he reported five new neutron-deficient isotopes of the element.[14] In his early co-ordination chemistry he studied the co-ordination chemistry of $[ReX_4]_n$ (X = Cl or Br) in a variety of complexes[109,116,140] with cyano,[107] oxo,[150,173,211] nitrido,[170] dithiocarbamato,[118,294] amine,[156] β-diketonate,[188] nitrile[235] and Schiff base[358] ligands. An important paper concerned binuclear carboxylato (particularly butyrato) complexes.[163] The complex *fac*-$Cs_2[ReO_3Cl_3]$ was made and the structure deduced from IR and Raman spectroscopy,[254] and the eight-co-ordinate dithiocarbamate complexes $[Re(S_2CNEt_2)_4]$-$[Re(CO)_3Cl(S_2CNEt_2)]$ were prepared, as was a molybdenum complex $[Mo(S_2CNEt_2)_4][ClO_4]$.[301]

The polyhydrido complexes $[ReH_7(PMe_3)_2]$, $[ReH_7(PMe_3)_2]{\cdot}NH_2Ph$ and $[Re_2H_8(PMe_3)_4]$ were isolated and characterised.[441] Reaction of $Re_2O_7$ with excess trimethylphosphine yields *trans*-$[ReO_2(PMe_3)_4][ReO_4]$; the crystal structure was obtained.[438] An unusual complex, made by reaction of the Schiff base bis(acetylacetone)ethylenediamine ($H_2L$) with $[ReOCl_3(PPh_3)_3]$, is $[Re_3O_4(L)][ReO_4]$; the crystal structure showed the presence of a near-linear O=Re–O–Re–O–Re=O backbone.[374]

## IV.5. Palladium, platinum and iridium chemistry

The zerovalent complex $[Pt(PPh_3)_3]$ reacts with $CS_2$ to give $[Pt(PPh_3)_2(CS_2)]$,[218] while reaction of $[Pt(PPh_3)_2(O_2)]$ with CO, $CO_2$ and $CS_2$ was shown to yield $[Pt(PPh_3)_2(CO_3)]$, $[Pt(PPh_3)_2(CO_4)]$ (a peroxocarbonate) and $[Pt(PPh_3)_2(O_2CS_2)]$ respectively in a remarkable paper.[274] The nature of 'iridium iodate' was studied and the polymeric formula $[Ir^{IV}\{I^{VII}O_3(OH)_2\}]_n$ assigned to the complex.

## IV.6. Phosphorus, antimony and tin trihalide complexes

As mentioned above Wilkinson's preparation in 1951[24] of $[Ni(PCl_3)_4]$ from $[Ni(CO)_4]$ and $PCl_3$ represented his entry into inorganic chemistry; he also made other species $[Ni(LX_3)_4]$ (L = P, X = F,[20] Br;[20] $LX_3$ = NCO[40] or NCS[408]) and the tricarbonyls $[Ni(CO)_3(SbCl_3)]$ and $[Fe(CO)_3(SbCl_3)_2]$.[21] Later work (1963) showed the first definitive evidence for $[SnCl_3]^-$ ligands with ruthenium, rhodium, iridium and platinum.[160,185]

## IV.7. Carboxylato, nitrosyl and cyano complexes

An early entry in this field was the preparation of $[Mo_2(OCOCH_3)_4]$, a very important complex of molybdenum in that it is a very useful precursor for other species, and also of course of much structural interest in its own right.[112] Wilkinson made a number of other carboxylates of molybdenum(II),[99,181] copper(II),[183] ruthenium, rhodium, palladium, platinum[182,178] and rhenium[206] complexes. The complexes $[Rh_2(OCOCH_3)_4]$, $[Ru(OCOCH_3)_4Cl]$ and $[Mo_2(OCOCH_3)_4]$ were found to function as homogeneous hydrogenation catalysts for alkenes and alkynes with dihydrogen when protonated in fluoroboric acid.[263]

Following the discovery of the trinuclear ruthenium carboxylates (see above) a number of other such species $[M_3O(OCOCH_3)_6(L)_3]^+$ were isolated [M = Cr, Mn, Fe, Co, Rh or Ir; L = pyridine (py) or methylpyridine].[303] Interesting work with osmium was also carried out: a series of osmium acetate complexes, *e.g.* $[Os(OCOCH_3)_3(PMe_3)_3]Cl$, $[Os_2(OCOCH_3)_4Cl(py)]$ were made,[404] and the crystal structure of the sky blue $K[OsO_2(OCOCH_3)_3]{\cdot}2MeCO_2H$ obtained. In the latter the two oxo ligands are *cis*, and there is one bidentate and two *trans* monodentate acetato ligands.[413]

His first papers on nitrosyl complexes date from 1956,[49,57] and then in a series of papers in collaboration with Jack Lewis a number of known and new nitrosyl complexes were made and sometimes reformulated, and the infrared distinction between '$NO^+$' and '$NO^-$' co-ordination used.[67,70,76,82,91,92,124,129]

Strictly speaking, cyano complexes should be considered as organometallic complexes since obviously they contain metal–carbon bonds. However they always seem to be considered as co-ordination complexes; the rigorous Gmelin classification admits them under this category. Wilkinson did much early work on cyano complexes: it may well be that part of his interest in nitrosyl, carbonyl and cyano complexes (and, later on, isocyano complexes) lay in the fact that they give sharp bands in an otherwise clear region of the infrared, *ca.* 1700–2200 $cm^{-1}$; in the early days infrared spectroscopy was one of the few physico-chemical techniques available to him. Certainly some of his students were interested in infrared (and Raman, a technique for which he was lamentably short of enthusiasm; he once described the IC instrument as being the 'instant incineration machine' after a number of his metal–metal bonded complexes were destroyed by the laser beam). He had a similarly jaundiced view of polarography, despite his use of the technique.[30,104,179,296]

He also did early work on the structures (as deduced from infrared) of metal–metal bonded cyano complexes,[77] on the first detection[81] by $^1H$ NMR spectroscopy of a metal–hydride complex in aqueous solution in $[CoH(CN)_5]^{3-}$, and then in $[RhH(CN)_5]^{3-}$,[98] work on rhenium cyano complexes,[80,104,107] and an acetylene cyano complex of cobalt.[90] Hydridocyano complexes of rhodium were later isolated.[202]

### IV.8. Miscellaneous aspects of co-ordination chemistry

There was some early work on platinum blue,[180] and with Dennis Evans—one of his earliest staff appointments to IC and a great and much-loved friend to so many of Wilkinson's collaborators—work on hydrogen bonding in anhydrous transition-metal cyanide free acids.[184] Infrared work was presented on complexes containing $[H_3O]^+$ and $[H_5O_2]^+$ cations.[172] Reaction of $[MoCl_3(THF)_3]$ with $PMe_3$ and sodium tetrahydroborate gave $[MoH(BH_4)(PMe_3)_4]$; the crystal structure was determined.[364]

Reaction of $[MoCl_4(THF)_2]$ with trimethylphosphine in tetrahydrofuran with magnesium as reductant gave $[MoH_2(PMe_3)_4]$, and this reacts with $CO_2$ to give, *via* an insertion reaction, $[MoH(OCOH)(PMe_3)_4]$. Single crystal X-ray studies of both complexes were carried out.[418] Reaction of lithium, sodium or potassium metals with $[W(OPh)_6]$ gave salts of $[W(OPh)_6]^-$; X-ray studies were made of the structures of the lithium and tetramethylammonium salts.[412] The neutral $[M(L)_3]$ complexes (M = Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, W or Re; L = 2,2′-bipyridyl or 1,10-phenanthroline) were made from the ligands and the metal chlorides in THF.[406]

## V. Use of Physical Techniques

Wilkinson was one of the first inorganic chemists to make use of whatever technique was available and appropriate; he was fortunate at Harvard in having access to the first NMR instruments (initially at DuPont but then later at Harvard), and he had the good sense and ability to consult and befriend experts at those or other institutions on various techniques. Thus in his early work he made creative use of $^1H$ NMR spectroscopy, as mentioned above for $[Re(\eta\text{-}C_5H_5)_2H]$[42] and many other systems; of broad-line $^1H$ NMR spectroscopy for studies on the inter-proton distances in $[FeH_2(CO)_4]$ with Sir Rex Richards in Oxford;[95] IR spectra for structural studies {*e.g.* an early use of group theoretical methods in carbonyl chemistry was made to determine the structures of $[Mn_2(CO)_{10}]$ and $[Re_2(CO)_{10}]$};[55] Raman spectroscopy (with Stammreich in São Paulo) for work on $[Fe_2(\eta\text{-}C_5H_5)_2(CO)_4]$;[75] calorimetry;[63,66,72] mass spectrometry;[47] polarography;[30,296] variable-temperature magnetochemistry[200] and circular dichroism.[175] Later of course single-crystal X-ray studies became of pre-eminent importance, and much of his later work relied heavily on the technique, but in the fifties and sixties it was often difficult, expensive and time-consuming to obtain such data. The first time that Wilkinson used the technique seems to be in 1979, when the crystal structures of $[Re_2O_3(S_2CNEt_2)_4]$ and $[ReN(S_2CNEt_2)_2]$ were determined.[277]

## VI. Alkyls and Aryls of the Transition Elements

Wilkinson's work on homoleptic alkyls and aryls of the transition elements was a major factor in overturning the view that had been current until the late sixties that such compounds were inherently unstable. First in 1970,[275] and later in his Nobel Prize address,[306,307] he pointed out that kinetic rather than thermodynamic factors are likely to be responsible for the failure to isolate simple transition metal–carbon σ-bonded compounds. His own work on $\eta^5$-cyclopentadienyl alkyls, and related studies on alkyl(carbonyl) and alkyl(tertiary phosphine) complexes, had clearly established the stabilising effect of the auxiliary ligands, which was generally held to be associated with their ability to form strong covalent bonds and to act as π acceptors; these features were believed to strengthen the metal–carbon σ bond.

One route by which many alkyls decompose is so-called β-elimination, in which a hydrogen atom migrates from the β-carbon atom of the alkyl chain to the metal to give an alkene–metal–hydride. Wilkinson, along with others, realised that the auxiliary ligands simply blocked co-ordination sites that would otherwise be available for the alkene and hydride ligands, thus explaining, for example, the remarkable stability of the ethylrhodium(III) cation $[Rh(C_2H_5)(NH_3)_5]^{2+}$.[241] He saw also that by use of alkyl groups that could not undergo β-elimination, such as $CH_2SiMe_3$, $CH_2CMe_3$, 1-adamantylmethyl, and even $CH_3$, it might be possible to prepare hitherto unknown alkyls of the transition metals, both with and without auxiliary ligands. This idea led to a notable series of papers in which such compounds were isolated from the reactions of simple transition-metal compounds, usually halides or carboxylates, with main-group alkyls, mainly of lithium, magnesium, zinc and aluminium, some of which were themselves prepared for the first time.[302,309,333] A selection of the transition element– and uranium–alkyls prepared by Wilkinson and his co-workers is given in Table 1. Notable are the range and structural diversity of the alkyls of rhenium, an element whose chemistry always fascinated him.

In addition to the usual problems involved in handling temperature- and air-sensitive compounds, the nature and yields of the reaction products often depended sensitively on the particular transition-metal compound and main-group reagents used, the solvent, and occasionally even the presence of small amounts of air. For example, reaction of chromium(III) chloride with the appropriate organolithiums gave stable, deep purple, tetrahedral organochromium(IV) compounds $[CrR_4]$ ($R = CH_2SiMe_3$, $CH_2CMe_3$ or $CH_2CMe_2Ph$),[275,283,287,293] presumably formed by oxidation of chromium(III) anions $[CrR_4]^-$. Even cyclohexyl, which is potentially capable of β-elimination, was shown to form a stable $[CrR_4]$ compound,[465] presumably because access to the metal is restricted by the sheath of bulky alkyl groups. Blockage of β-elimination in the alkyl groups allowed other possible breakdown pathways to be observed, mainly in the case of early 4d- and 5d-elements. Complexes containing either terminal or bridging trimethylsilylmethylidyne, $CSiMe_3$, were formed by deprotonation at the α position of $CH_2SiMe_3$, *e.g.* $[M^V_2(\mu\text{-}CSiMe_3)_2R_4]$ (M = Nb,[284] Ta,[284] W[328] or Re[376,475]), $[Re^{VII}(CSiMe_3)ClR_3]$[475] and $[M^{VI}(CSiMe_3)R_3]$ (M = Mo[398] or W[398]) ($R = CH_2SiMe_3$). In contrast, with tertiary phosphine complexes of ruthenium, osmium and rhodium, γ-elimination of $SiMe_3$ to form a pair of $CH_2SiMe_3$ groups was a common feature, *e.g.* the formation of $[Os\{(CH_2)_2SiMe_2\}(PMe_3)_4]$ from $[Os(O_2CMe)_2(PMe_3)_4]$ and $[Mg(CH_2SiMe_3)_2]$.[429] In the notable instance of the reaction of $[Ru_3O(O_2CMe)_6(H_2O)_3][O_2CMe]$ with dimethylmagnesium and trimethylphosphine, the methyl groups were deprotonated to give, among other products, the confacial bioctahedral tri-μ-methylenediruthenium(III) complex $[Ru_2(\mu\text{-}CH_2)_3(PMe_3)_6]$.[348,363] Acid treatment of the reaction mixture afforded a trinuclear, mixed-valence cation $[(Me_3P)_4Ru^{III}(\mu\text{-}CH_2)_2Ru^{IV}(\mu\text{-}CH_2)_2Ru^{III}(PMe_3)_4]^{2+}$, in which the central metal atom is bound only to methylene groups in a tetrahedral arrangement.[377]

One of Wilkinson's finest achievements was surely the isolation and characterisation of the volatile alkyls and oxoalkyls of the 4d- and 5d-elements in high oxidation states. As he pointed out,[299] the discovery of the volatile hexamethyls of tungsten and rhenium 'can be said to dispose of the view that the highest oxidation states of the transition elements are accessible only when the metal atom is bound to electronegative atoms'. The yields of $[WMe_6]$, a red solid, obtained initially from $[WCl_6]$ and 3 equivalents of methyllithium[286,299] were evidently erratic (the necessity for traces of oxygen was claimed), but it was found later that use of 6 equivalents of trimethylaluminium provided a more reliable procedure.[314,329] Reaction of methyllithium with $[ReOCl_3(PPh_3)_2]$ or $[ReOCl_4]$ and subsequent work-up in the presence of traces of air or with $H_2O_2$ gave red-purple oxotetramethylrhenium, $[ReOMe_4]$,[304,311] which, on treatment with trimethylaluminium, gave green $[ReMe_6]$.[314,324] Given their large, positive enthalpy of formation,[325] it is perhaps not surprising that these permethyl compounds tended to detonate unpredictably in the

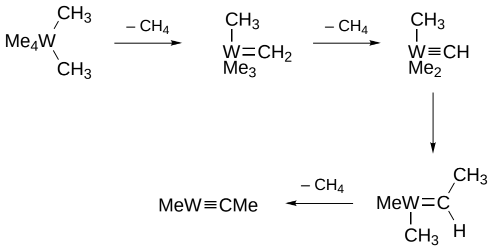
**Table 1** Selected transition-metal alkyls made by Wilkinson *et al.**

| Formula of complex | Alkyl group (R) | Ref. |
|---|---|---|
| Vanadium | | |
| $[\mathrm{V^{V}(O)R_3}]$ | $\mathrm{CH_2SiMe_3}$ | 275, 283 |
| $[\mathrm{V^{IV}R_4}]$ | $\mathrm{CH_2SiMe_3}$, adme | 275, 283, 380 |
| Chromium | | |
| $[\mathrm{Cr^{IV}R_4}]$ | Me, $\mathrm{CH_2SiMe_3}$, $\mathrm{CH_2CMe_3}$, $\mathrm{CH_2CMe_2Ph}$, $\mathrm{CH_2CPh_3}$, adme | 275, 283, 287, 293, 380 |
| $[\mathrm{Cr^{II}{}_2R_2(\mu\text{-}R)_2(PMe_3)_2}]$ | $\mathrm{CH_2SiMe_3}$ | 339, 344 |
| Manganese | | |
| $[\mathrm{Mn^{II}R_2}]_n$ | $\mathrm{CH_2SiMe_3}$ ($n = \infty$), $\mathrm{CH_2CMe_3}$ ($n = 4$), $\mathrm{CMe_2Ph}$ ($n = 2$), adme ($n = 1$) | 319, 327, 380 |
| $[\mathrm{Li(tmen)}]_2[\mathrm{Mn^{II}R_4}]$ | Me | 319, 327 |
| $[\mathrm{Mn^{II}{}_2R_2(\mu\text{-}R)_2(PMe_3)_2}]$ | $\mathrm{CH_2SiMe_3}$, $\mathrm{CH_2CMe_3}$, $\mathrm{CH_2Ph}$ | 411, 419 |
| $[\mathrm{Mn^{II}R_2(PMe_3)_2}]$ | $\mathrm{CH_2CMe_2Ph}$ | 421, 426 |
| $[\mathrm{Mn^{II}R_2(dmpe)}]$ | $\mathrm{CH_2CMe_2Ph}$, $\mathrm{Bu^t}$ | 426, 442 |
| $[\mathrm{Mn^{II}{}_2R_2(\mu\text{-}R)_2(\mu\text{-}dmpe)}]$ | $\mathrm{C_6H_{11}}$ | 442 |
| $[\mathrm{Mn^{IV}R_4(dmpe)}]$ | Me | 424 |
| Cobalt | | |
| $[\mathrm{Li(tmen)}]_2[\mathrm{Co^{II}R_4}]$ | Me, $\mathrm{CH_2SiMe_3}$ | 319, 495 |
| $[\mathrm{Co^{II}R_2(tmen)}]$ | $\mathrm{CH_2CMe_3}$, $\mathrm{CH_2SiMe_3}$ | 319, 495 |
| $[\mathrm{Li(tmen)_2}][\mathrm{Co^{II}ClR_2}]$ | $\mathrm{CH(SiMe_3)_2}$ | 495 |
| Molybdenum | | |
| $[\mathrm{Mg(THF)_4}][\mathrm{Mo^{V}(O)R_4}]_2$ | Me | 470 |
| $[\mathrm{Mo^{IV}ClR_3(PMe_3)}]$ | $\mathrm{CH_2SiMe_3}$ | 354, 365 |
| $[\mathrm{Mo^{III}{}_2R_6}]$ | $\mathrm{CH_2CMe_3}$, $\mathrm{CH_2SiMe_3}$ | 282, 283, 339, 344, 398 |
| $[\mathrm{Mo^{II}{}_2R_4(PMe_3)_4}]$ | Me | 344 |
| $\mathrm{Li_4[Mo^{II}{}_2R_8]\cdot 4Et_2O}$ | Me | 305 |
| Tungsten | | |
| $[\mathrm{W^{VI}R_6}]$ | Me | 286, 299, 314, 315, 318, 325, 329, 351, 393 |
| $[\mathrm{Mg(THF)_4}][\mathrm{W^{V}(O)R_4}]_2$ | Me | 470 |
| $[\mathrm{W^{III}{}_2R_6}]$ | $\mathrm{CH_2SiMe_3}$ | 283, 328 |
| Rhenium | | |
| *cis*-$[\mathrm{Re^{VII}(O)_2R_3}]$ | Me | 324 |
| $[\mathrm{Re^{VI}R_6}]$ | Me | 323, 324, 351 |
| $\mathrm{Li_2[Re^{VI}R_8]\cdot tmen}$ | Me | 323, 324 |
| $[\mathrm{Re^{VI}(O)R_4}]$ | Me, $\mathrm{CH_2SiMe_3}$ | 304, 311, 312, 351 |
| $[\mathrm{Re^{VI}{}_2O_3R_6}]$ | Me, $\mathrm{CH_2SiMe_3}$ | 311, 448 |
| $[\mathrm{Mg(THF)_4}][\mathrm{Re^{V}(O)R_4}]_2$ | Me, $\mathrm{CH_2SiMe_3}$ | 448, 470 |
| $[\mathrm{Re^{IV}{}_2R_8(\mu\text{-}N_2)}]$ | $\mathrm{CH_2SiMe_3}$, $\mathrm{CH_2CMe_2Ph}$ | 343 |
| $[\mathrm{Re^{III}{}_3R_6Cl_3}]$ and its derivatives | Me, $\mathrm{CH_2CMe_3}$, $\mathrm{CH_2SiMe_3}$, $\mathrm{CH_2Ph}$ | 335, 343, 385, 388, 477 |
| $[\mathrm{Re^{IV}{}_3R_{12}}]$ | $\mathrm{CH_2SiMe_3}$ | 343 |
| $\mathrm{Li_2[Re^{III}{}_2R_8]\cdot 2Et_2O}$ | Me | 330 |
| Ruthenium | | |
| $[\mathrm{Ru^{V}{}_2(O)_2R_6}]$ | $\mathrm{CH_2CMe_3}$, $\mathrm{CH_2SiMe_3}$ | 437, 461 |
| $[\mathrm{Ru^{III}{}_2R_6}]$ | $\mathrm{CH_2CMe_3}$, $\mathrm{CH_2SiMe_3}$ | 437, 461 |
| $[\mathrm{Li(tmen)}]_3[\mathrm{Ru^{III}R_6}]$ | Me | 499 |
| Osmium | | |
| $[\mathrm{Os^{VI}(O)R_4}]$ | $\mathrm{CH_2SiMe_3}$ | 403 |
| $[\mathrm{Os^{III}{}_2(O_2CMe)_2R_4}]$ | $\mathrm{CH_2CMe_3}$ | 437, 461 |
| Rhodium, Iridium | | |
| $[\mathrm{Rh^{IV}{}_2(\mu\text{-}O)R_6}]$ | $\mathrm{CH_2CMe_3}$ | 493, 499 |
| $[\mathrm{Li(tmen)}]_3[\mathrm{M^{III}R_6}]$ (M = Rh or Ir) | Me | 493, 499 |
| Uranium | | |
| $\mathrm{Li_3[U^{V}R_8]\cdot 3diox}$ | Me, $\mathrm{CH_2CMe_3}$, $\mathrm{CH_2SiMe_3}$ | 319, 334 |
| $\mathrm{Li_2[U^{IV}R_6]\cdot 8Et_2O}$ | Me, $\mathrm{CH_2SiMe_3}$ | 319, 334 |

* Abbreviations: adme = 1-adamantylmethyl; tmen = tetramethylethane-1,2-diamine; dmpe = 1,2-bis(dimethylphosphino)ethane; diox = 1,4-dioxane.

solid state, even in the absence of air, or on vacuum sublimation.[314,318,351] Analysis of the ESR spectrum of $[\mathrm{ReMe_6}]$ suggested a distorted octahedral geometry,[323] but the fact, now known from electron diffraction of the vapour and crystal-structure analysis, that $[\mathrm{WMe_6}]$ is trigonal prismatic was not suspected.

Wilkinson showed that $[\mathrm{WMe_6}]$ and $[\mathrm{ReMe_6}]$ are co-ordinatively unsaturated. Both compounds form 'ate' complexes with methyllithium, $\mathrm{Li_2[MMe_8]}$ (M = W or Re)[314,324,329] and $[\mathrm{WMe_6}]$ forms a red 1 : 1 adduct with $\mathrm{PMe_3}$. Irradiation of the latter with an excess of $\mathrm{PMe_3}$ gave the methyl(ethylidyne) compound *trans*-$[\mathrm{WMe(CMe)(PMe_3)_4}]$ *via* successive eliminations of methane, as shown in Scheme 1.[393] One can speculate that similar steps occur in the spontaneous decomposition of $[\mathrm{WMe_6}]$.



**Scheme 1** Proposed mechanism for the decomposition of [WMe$_6$] (PMe$_3$ ligands omitted for clarity)

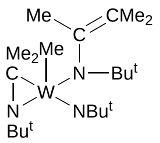
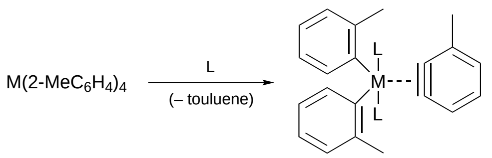


**Fig. 5** Reaction product of [WMe$_6$] with *tert*-butyl isocyanide

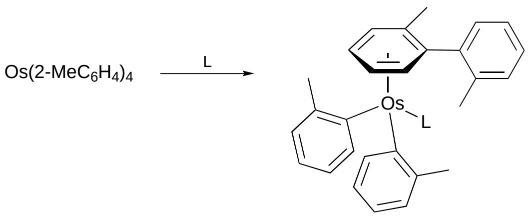
Wilkinson also showed that diamagnetic [WMe$_6$] and paramagnetic [ReOMe$_4$] differ greatly in their behaviour with nitric oxide. The tungsten compound gave an eight-co-ordinate complex [WMe$_4${ON(Me)NO}$_2$] containing bidentate *N*-methyl-*N*-nitrosohydroxylaminato ligands,[290] whereas the rhenium compound underwent elimination of dimethyldiazene to give *cis*-[ReO$_2$Me$_3$].[314,324] This work led to detailed studies of the reactions of transition-metal alkyls with nitric oxide.[381,395] In the reaction of [WMe$_6$] with *tert*-butyl isocyanide, five of the six available methyl groups were found to transfer to the ligand, giving the complex shown in Fig. 5.[391,397]

Although no other neutral transition-metal hexamethyls are known, Wilkinson was able to synthesise the trianions [MMe$_6$]$^{3-}$ of rhodium(III), iridium(III) and ruthenium(III), in the form of their very air-sensitive [Li(tmen)]$^+$ salts.[493,499] From the reaction of [RhCl$_3$(tht)$_3$] (tht = tetrahydrothiophene) and neopentyllithium or neopentylmagnesium bromide in the presence of Me$_3$NO or of traces of dioxygen, he obtained the oxo-bridged dirhodium(IV) compound [Rh$_2$(μ-O)(CH$_2$CMe$_3$)$_6$].[499] The first homoleptic alkyls of a platinum metal, [Ru$_2$R$_6$] (R = CH$_2$CMe$_3$ or CH$_2$SiMe$_3$), were also isolated and shown to consist of a staggered, ethane-like arrangement of two Ru$^{III}$R$_3$ fragments joined by an unsupported metal–metal bond.[437,461] These compounds reacted with nitrosobenzene to give dioxodiruthenium(V) complexes, [Ru$_2$(O)$_2$R$_6$], whose oxo groups could be transferred to propene to give methyloxirane. The existence of these remarkable compounds demonstrated that even the platinum metals could be sustained in high oxidation states by ligation of a suitable alkyl group.

Wilkinson's work on σ-aryls began with phenyl and 2-methoxyphenyl derivatives, usually containing PMe$_3$ as co-ligand, of molybdenum, rhenium, ruthenium and rhodium.[355] However, by use of sterically hindered aryl groups, such as 2-tolyl, 2,5-xylyl and mesityl (mes), he was able to prepare a family of homoleptic σ-aryls, mainly of the 4d- and 5d-transition elements, most of whose members have no σ-alkyl counterparts. Among them are the tetrakis(aryls), [MR$_4$] (M = Cr, Mo, W,[488,501] Re,[476,488] Ru,[476,524] Os[454,465] or Ir[511,524]), in which the metal atoms are co-ordinated tetrahedrally; this geometry was unprecedented for compounds of the 4d- and 5d-elements in the +4 oxidation state. It was even possible to prepare tetraphenylosmium(IV), [OsPh$_4$], and, for ruthenium(IV) and osmium(IV), the tetrakis(cyclohexyl), [M(C$_6$H$_{11}$)$_4$].[454,465] The ruthenium and iridium complexes [M(mes)$_4$] (M = Ru or Ir)[524] and the osmium complex [Os(2-MeC$_6$H$_4$)$_4$][485] could be oxidised reversibly to give isolable salts of the corresponding metal(V) cations [M(aryl)$_4$]$^+$, in which tetrahedral co-ordination is maintained. Reaction of (mes)MgBr with [MCl$_3$(tht)$_3$] (M = Rh or Ir) gave ligand-free tris(mesityl) compounds, [M$^{III}$(mes)$_3$],[502,515,524] which are pyramidal in the solid state but may be planar in solution. Both [Ir$^{III}$(mes)$_3$] and [Ir$^{IV}$(mes)$_4$] reacted with dioxygen to give an oxoiridium(V) complex [Ir(O)(mes)$_3$].[531] A dioxoosmium(VI) complex [OsO$_2$(mes)$_2$] was also obtained.[459]



**Scheme 2** Dihapto-aryne complexes (M = Nb, Ta, Mo, W or Re; L = PMe$_2$Ph or PMe$_3$)



**Scheme 3** Product of coupling two σ-aryl fragments in homoleptic aryl complexes (L = PMe$_3$ or CO)

Reaction of *mer*-[IrCl$_3$(SEt$_2$)$_3$] with an excess of [Mg(mes)$_2$(THF)$_2$] gave paramagnetic *trans*-[Ir(mes)$_2$(SEt$_2$)$_2$], a very rare example of a planar iridium(II) complex. The sulfide ligands could be replaced by tertiary phosphines, and the derivative *trans*-[Ir(mes)$_2$(PMe$_3$)$_2$] reacted with nitric oxide to give an iridium(III) complex [Ir(NO)(mes)$_2$(PMe$_3$)$_2$] containing a bent nitrosyl group.[523] Attempts to prepare the rhodium(II) analogue of *trans*-[Ir(mes)$_2$(SEt$_2$)$_2$] gave only [Rh(mes)$_3$], but by use of the even more bulky aryl group 2,4,6-triisopropylphenyl the planar, paramagnetic rhodium(II) complex [Rh(2,4,6-Pr$^i_3$-C$_6$H$_2$)$_2$(tht)$_2$] was isolated and structurally characterised.[514] The tris(mesityl)nickel(II) anion, [Ni(mes)$_3$]$^-$, was obtained as its [Li(THF)]$^+$ complex and shown to be T-shaped with agostic Ni···CH$_3$ interactions.[550]

The niobium and tantalum compounds [MR$_4$] (M = Nb or Ta; R = 2-MeC$_6$H$_4$ or 2,5-Me$_2$C$_6$H$_3$) could not be isolated but were shown to react *in situ* with PMe$_2$Ph to give dihapto-aryne complexes as shown in Scheme 2;[516] similar reactions occurred when the 2-tolyls of rhenium,[482,484] molybdenum[501] and tungsten[501] were treated with tertiary phosphines. The rhenium 3-methylbenzyne complex [Re(2-MeC$_6$H$_4$)$_2$(η$^2$-3-MeC$_6$H$_3$)(PMe$_2$Ph)$_2$] underwent reversible one-electron oxidation, *e.g.* with [Fe(η-C$_5$H$_5$)$_2$]$^+$ or I$_2$, to the corresponding cation.[484] The abstraction of an *ortho*-hydrogen atom from one aryl group by another to give free arene and dihapto-aryne appears to be a characteristic reaction of aryls of the early 4d- and 5d-elements. In contrast, addition of PMe$_3$ or CO to [Os(2-MeC$_6$H$_4$)$_4$] induced coupling of two of the 2-tolyl groups to form the η$^6$-2,2′-bitolyl complexes represented in Scheme 3.[482,487,547] This σ-to-π conversion bears some similarity to processes supposed to occur in the classic Hein reaction in which bis(arene)chromium cations are formed *via* triphenylchromium(III) from CrCl$_3$ and PhMgBr.

Wilkinson's work on homoleptic alkyls and aryls of the transition metals was incorporated into two general reviews.[505,543]

## VII. The Last Fifteen Years

In the last fifteen years of his research career, in addition to the continuing study of σ-metal–carbon bond-containing complexes, Wilkinson investigated several areas of co-ordination chemistry including: (*i*) the use of strongly basic and/or chelating small phosphines for stabilisation of electron-rich transition-metal complexes and the study of their reactivity

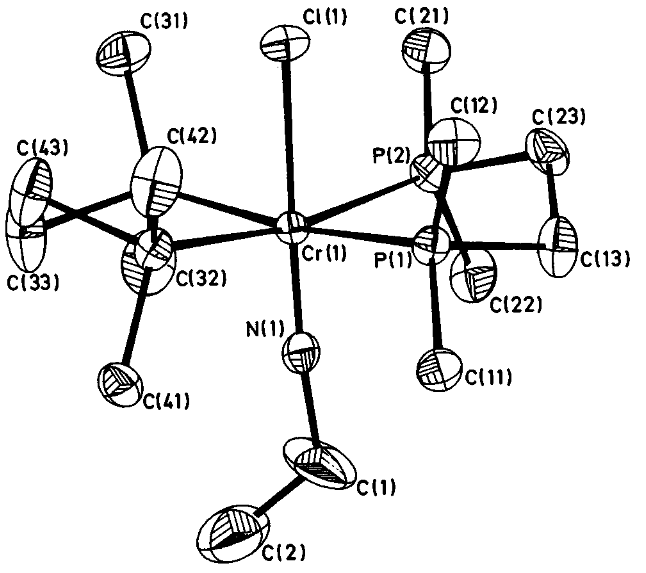


**Fig. 6** Structure of the cation $[CrCl(NEt)(dmpe)_2]^+$ (ref. 469)

with small molecules especially $H_2$, organic nitriles, $CO_2$, CO; (*ii*) the chemistry of hydride and aluminohydride complexes of transition metals; (*iii*) the chemistry of amido and alkoxide complexes of early and high oxidation state metals and the analogies between oxo and imido complexes as well as the reactivity of the latter.

In all these areas he produced seminal papers, some of them describing spectacular compounds—'textbook cases' as he used to claim—and unusual reactions. His ability to detect lacunae in the chemical literature, to formulate problems guided by empirical analogy and maximum simplicity and finally to propose imaginative and brave hypotheses in order to account for observed experimental facts are still very apparent in this last period.

### VII.1. Phosphine and related complexes

Wilkinson's long interest in metal phosphine complexes is reflected in this period by the use of trimethylphosphine, dimethylphosphinomethane (dmpm) and dimethylphosphinoethane (dmpe) ligands for the stabilisation of unusual metal environments and promoting unexpected reactivity. This work gave rise to $Fe^{II,0}$, $Ru^{II,0}$,[439] homo- and hetero-dinuclear iron/chromium[446] dimethylphosphinomethane complexes and alkyls and hydrides derived from them. Dimethylphosphinoethane complexes of $Cr^{IV}$ *e.g.* $[CrH_4(dmpe)_2]$, $Cr^0$ *e.g.* *cis*-$[Cr(N_2)_2(dmpe)_2]$ and *trans*-$[Cr(CO)_2(dmpe)_2]$,[440] $Cr^I$ *e.g.* *trans*-$[Cr(CO)_2(dmpe)_2][BPh_4]$ and $Cr^{II}$ *e.g.* *trans*-$[Cr(MeCN)_2(dmpe)_2][CF_3SO_3]_2$[456] were fully characterised. The latter raised the issue of reactivity of acetonitrile co-ordinated to electron-rich metals. Facile protonation with methanol produced ethylimido *trans*-$[Cr^{IV}Cl(NEt)(dmpe)_2][CF_3SO_3]$, shown in Fig. 6, and ethylideneamido *trans*-$[Cr^{IV}(N{=}CHMe)_2(dmpe)_2][BPh_4]_2$, the first structurally characterised octahedral $Cr^{IV}$ compounds.[469] Similar chemistry was developed for molybdenum.[483] Protonation of the co-ordinated acetonitrile in $[Mo(\eta^5\text{-}C_5H_5)_2(\eta^2\text{-}MeCN)]$ giving rise to $[Mo(\eta^5\text{-}C_5H_5)_2(MeCN)(HN{=}CHMe)][BF_4]_2$ was undertaken to study the generality of the reaction.[478] Dimethylphosphinoethane was also used to stabilise alkyls, hydrides and aluminohydrides of manganese-(II) and -(IV),[421,424,442] titanium, vanadium, chromium, iron and rhenium.[445]

### VII.2. Hydride and aluminohydride complexes

Continued interest in the hydrides of the transition metals resulted in the development of synthetic methods for Group VI polyhydrides stabilised by $PMe_3$. The complex $[WH_6(PMe_3)_3]$ was prepared from $[WCl_4(PMe_3)_3]$ and lithium aluminium hydride as reductant in the presence of $H_2$.[418,441,425] The complex $[MoH_2(PMe_3)_5]$ was prepared by reduction of $[MoCl_4(THF)_2]$ with Mg in the presence of $H_2$.[425] Insertion reactions of $CO_2$ and PhNCO into the Mo–H bonds gave rise to $[MoH(CO_2H)(PMe_3)_4]$ and $[MoH\{PhNC(H)O\}(PMe_3)_4]$ respectively which were characterised by X-ray and spectroscopic methods.[425] Reduction of $[Re(NPh)Cl_3(PMe_3)_2]$ with lithium aluminium hydride gave rise to $[ReH_7(PMe_3)_2]\cdot NH_2Ph$.[441] Careful study of the reduction of $[WCl_4(PMe_3)_3]$ with lithium aluminium hydride opened a new area of chemistry involving aluminohydride as ligand in unprecedented co-ordination modes.[449] Aluminohydrides of chromium,[455] molybdenum,[455] tungsten,[451,455,457,464] rhenium,[451] ruthenium and osmium[453] were characterised by spectroscopic methods and in many cases diffraction methods. A substantial part of this chemistry has been described in a review.[463]

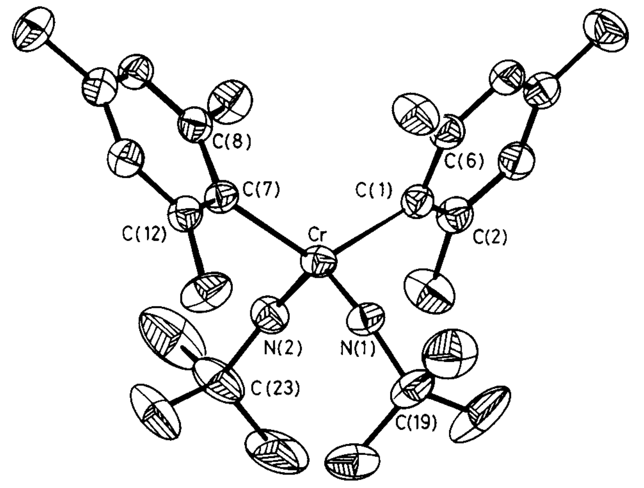


**Fig. 7** Structure of $[Cr(mes)_2(NBu^t)_2]$ (ref. 474)

### VII.3. Imido, amido and alkoxide complexes

Wilkinson's first approach to the chemistry of high oxidation state imido (RN=) and nitrido (N≡) ligands probably arose from his involvement in the study of oxo-, oxo-alkyl- and oxo-aryl-complexes (*cf.* oxo-alkyls and -aryls of rhenium and osmium[448,459,470,479]) and attempts to investigate analogous isoelectronic imido and nitrido species. As a result of these studies it turned out that the imido group in high oxidation complexes can act as a good 'spectator' ligand, giving thermally stable, less reducible compounds.

The first papers Wilkinson published in this area described dithiocarbamate derivatives of $[Re^V(NPh)Cl_3(PPh_3)_2]$[285] which was reported earlier by Chatt, and the crystal structure of $[ReN(S_2CNEt_2)_2]$. Additional work with $[Re^V(NPh)Cl_3(PMe_3)_2]$ gave rise to imido alkyls $[Re(NPh)R_3(PMe_3)_2]$[401] while reduction under a variety of conditions produced amido–dinitrogen $[Re(NHPh)(N_2)(PMe_3)_4]$, the rare amido–hydride $[Re(NHPh)(H)\{(\eta^2\text{-}CH_2PMe_2)\}(PMe_3)_4]$, imido–diene $[Re(NHPh)(\eta^4\text{-}C_4H_6)(PMe_3)_2]$[402] and the $CO_2$ complex $[Re(NHPh)(\eta^2\text{-}CO_2)(PMe_3)_3]$.[409] The use of *tert*-butylimido groups to stabilise chromium(VI)–aryl bonds, previously regarded as unstable, resulted in the isolation of the stable $[Cr(Bu^tN)_2(mes)_2]$,[474] depicted in Fig. 7. The reactivity of the Cr–C bonds towards insertion in this complex as well as analogous molybdenum chemistry were described in the same paper.

The isoelectronic relationship of $(RN)_2Cr^{VI}$ and $(\eta\text{-}C_5H_5)_2Ti^{IV}$ fragments and the obvious interest in the catalytic chemistry of $Cr^{VI}$ related to polymerisation of alkenes intensified efforts to discover a general entry to $Cr^{VI}$–imido chemistry, analogous to bis(cyclopentadienyl) halides of titanium and zirconium. This led to the synthesis of $[Cr(Bu^tN)_2Cl_2]$,[503] which, since its discovery, has been used repeatedly for the preparation

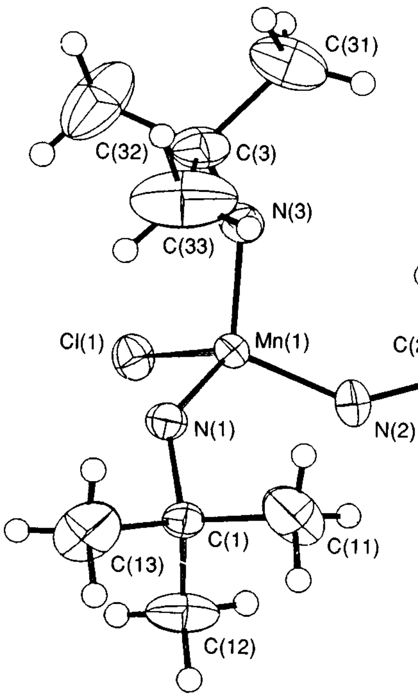


**Fig. 8** Structure of $[\mathrm{MnCl(NBu^t)_3}]$ (ref. 529)

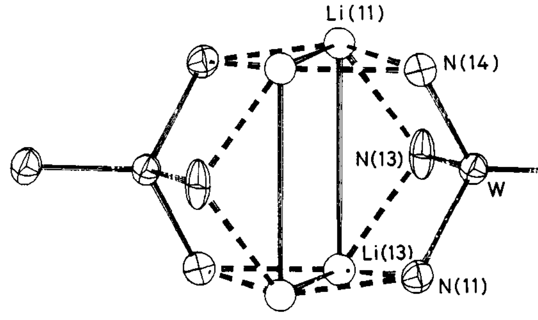


**Fig. 9** Structure of $\mathrm{Li_4[W_2(NBu^t)_8]}$ (ref. 489)

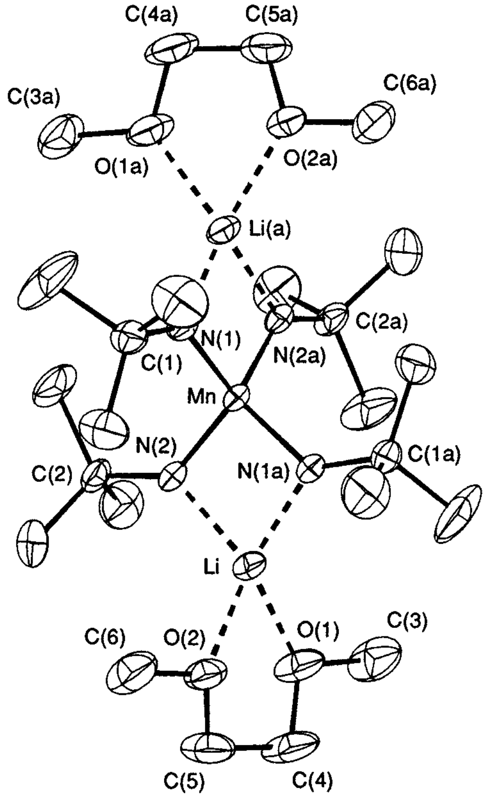


**Fig. 10** Structure of $\mathrm{[Li(dme)]_2[Mn(NBu^t)_4]}$ (ref. 536)

of numerous imido complexes of chromium. In a later paper a simple synthesis of arylimido chromium complexes *via* isocyanate exchange was described together with many derivatives of arylimido species.[544] Of major interest in the chromium–imido chemistry is the synthesis of stable neutral imido compounds of chromium(V), $[\mathrm{Cr(Bu^tN)Cl_3L_2}]$, by chlorination of $[\mathrm{Cr(Bu^tN)_2Cl_2}]$ and other similar compounds of this unusual oxidation state, bearing the $\mathrm{Bu^tN{=}Cr^V}$ fragment.[513] Some fifty crystal structures of chromium–imido derivatives were determined.

The idea of using imido groups to stabilise unusual high oxidation state compounds was also explored with rhenium, where numerous alkyls, aryls, amides, phosphides and related imido derivatives were synthesised and studied[479,491,508,510,517] as well as those of osmium.[496] Work on imido complexes of ruthenium gave two remarkable compounds: the blue *trans*-$[\mathrm{Ru(2,6\text{-}Pr^i_2C_6H_3N)_2(PMe_3)_2}]$[525] and the red-brown cyclohexylamido $[\mathrm{Ru^{IV}\{NH(C_6H_{11})\}\{OCEt(R)(O)O\}_2}]^-$ which was oxidised with dioxygen to the corresponding paramagnetic ruthenium(V) imido complex.[522] Both compounds are rare examples of high oxidation state ruthenium imido species.

Probably the highlight of Wilkinson's high oxidation state chemistry is the fortuitous synthesis of the spectacularly thermally stable $[\mathrm{Mn^{VII}(Bu^tN)_3Cl}]$, Fig. 8, which opened a new era in high oxidation state manganese chemistry.[529] Wilkinson, with his characteristic enthusiasm for significant new compounds was heard to claim that 'this discovery is the most important development in high oxidation state chemistry of manganese for over 150 years' (*i.e.* since Glauber described the permanganate anion). Numerous manganese–imido complexes in oxidation states (V) and (VII) followed, including thiolates, acetates, amides, alkyls[536,540,541] and the first and only example of a $\mathrm{Mn^{VII}{-}C}$ bond in $[\mathrm{Mn(Bu^tN)_3(C_6F_5)}]$. The addition of approximately thirty outstanding new compounds in an area of chemistry which had been dominated by $\mathrm{MnO_4^-}$ and $\mathrm{MnO_4^{2-}}$ demonstrate the impact of this discovery.

During his early involvement in imido chemistry he noticed in the literature the absence of 'homoleptic' imido compounds analogous to the well established oxo species. Attempts to prepare imido analogues of $\mathrm{MO_4^{2-}}$ (M = Cr, Mo or W) resulted in isolation of the complexes $\mathrm{Li_2[M(NBu^t)_4]}$. The tungsten derivative was structurally characterised, as shown in Fig. 9,[489] and its reactivity was studied in detail giving rise to interesting heterometallic imido complexes with metallic (Al, Ga or Cu)[498] or main-group species ($\mathrm{PX_3}$,[539] PhSCl[542] or PhSeBr[548]). Also during the same period the two important homoleptic imido complexes were prepared: $[\mathrm{Re^{VI}(NBu^t)_2(\mu\text{-}NBu^t)]_2}$[491] and the imido analogue of perrhenate $[\mathrm{(tmen)Li][Re(NBu^t)_4}]$.[489,491] The former was used to prepare heterometallic imido clusters.[551] The homoleptic imido complexes of manganese, $[\mathrm{Mn^{VI}(NBu^t)_2(\mu\text{-}NBu^t)]_2}$, $[\mathrm{Mn\{(Bu^tN)_2Mn(\mu\text{-}NBu^t)_2\}_2}]^{2+}$ and $\mathrm{[Li(dme)]_2[Mn(NBu^t)_4]}$ (dme = 1,2-dimethoxyethane), shown in Fig. 10, were obtained by reduction of $[\mathrm{Mn(NBu^t)_2Cl}]$ under specific conditions.[536,541] Finally, the homoleptic volatile imido complex of osmium(VIII), $[\mathrm{Os(NBu^t)_4}]$ was prepared from $[\mathrm{OsO_4}]$[503] and its structure in the gas phase as determined by electron diffraction was described in a joint paper.[537] By reduction of $[\mathrm{Os(NBu^t)_4}]$, pictured in Fig. 11, dimeric homoleptic imido compounds of osmium, *e.g.* $[\mathrm{Os(NBu^t)_2(\mu\text{-}NBu^t)]_2}$, also followed.[503,512]

Reactivity studies of the co-ordinated imido groups likewise date from this period. In addition to the reactions of the homoleptic imido complexes already mentioned, Wilkinson investigated the insertion of isocyanates into amido and imido groups giving ureato- or biuretato-complexes,[526,515] insertion of azides and isocyanides to imido groups,[546,552] and imido transfers to isocyanides[545] and *tert*-butylimido decompositions to nitrido ligands.[515]

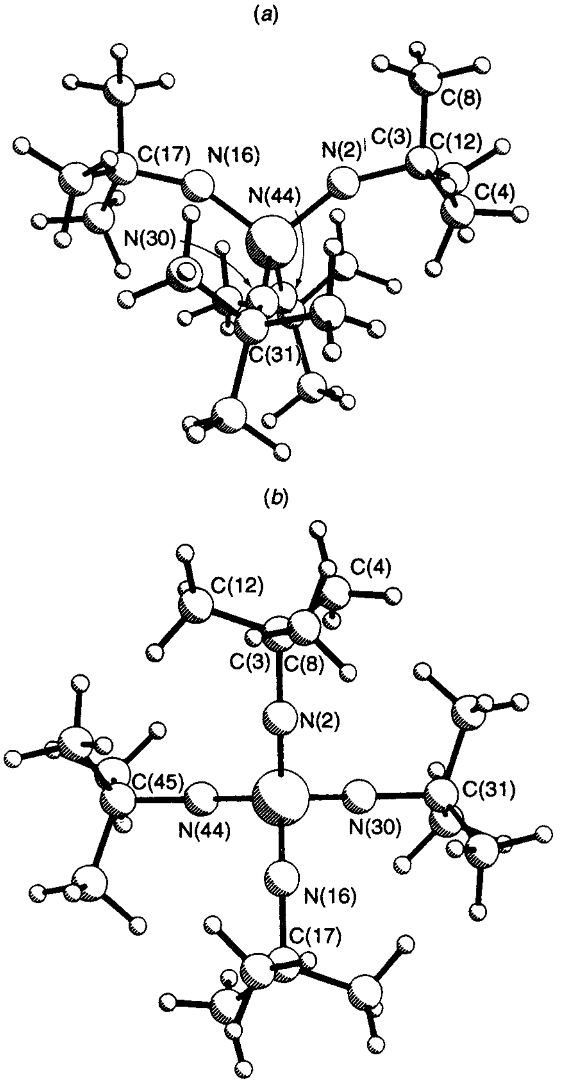


**Fig. 11** Two views of $[Os(NBu^t)_4]$ (ref. 537)

Complementary to the amide and alkoxide complexes that were studied in relation to imido chemistry (see above), Wilkinson initiated studies in high oxidation state alkoxide and amide oxo complexes of rhenium and chromium,[530] areas that are still not fully investigated due to the experimental difficulties they present. An improved method for the preparation of $[Re^{VI}OCl_4]$ by oxidation of $ReCl_5$ with dioxygen was reported together with the monomeric and dimeric alkoxides and amides derived from it.[383] An analogous method for the synthesis of $[Re^{VII}O_3Cl]$ by combustion of $[Re^{III}{}_3Cl_9]$ with dioxygen followed.[432] Monomeric amides and alkoxides of type $ReO_3X$ ($X = NR_2$ or OR) were also included. This paper appeared with a cautionary note, since on one occasion during the preparation of $[Re^{VII}O_3Cl]$ a violent explosion (possibly due to oxides of chlorine) and fire ensued sending Peter Edwards (now Professor) to the hospital. Wilkinson insisted on fighting the fire personally (having forcibly pushed one of his students aside in order to do so). To Wilkinson's relief the replacement cost of the completely destroyed fume-cupboard was covered by insurance.

*triangulo*-Trirhenium(iii) alkoxides soon followed and some of them were structurally characterised.[481] Also during this period a series of papers appeared dealing with the *o*-phenylenediamido(2−) and *o*-amidobenzenethiolato(2−) ligands. Homoleptic complexes of rhenium-(vii), -(vi), -(v) and osmium(vi) were spectroscopically and electrochemically studied.[490] More work with tungsten, chromium, vanadium and manganese ensued as well.[520,521] Functionalised six-electron donor chelating amides and sulfenamides as alternatives to cyclopentadienyls were prepared and their complexes with early transition metals studied.[549,548,553] Finally, Wilkinson studied the ligand properties of bulky alkoxides, [bis(*tert*-butyl)-methoxides and adamantoxides]. Monomeric, neutral and anionic homoleptic complexes of first-row metals, niobium and molybdenum[373,378,380] were isolated.

In conclusion, we have attempted to illustrate the enormous range of areas in which Wilkinson's reseach has made substantial contributions. This outstanding body of work clearly demonstrates one of Wilkinson's dearest beliefs, namely that innovative and imaginative exploratory synthesis can be one of the most creative approaches for the discovery of new chemistry. Furthermore, all of this research was 'blue skies' and curiosity driven. He had many close, and successful, interactions with industry over the years, but the work in his research group was virtually never directed towards immediate short term applications.

## Acknowledgements

Much of the material on Wilkinson's early life has been obtained from conversations with, and also a written memoir from, his brother John, and from an audio tape made at a lecture, 'Some Recollections', given by Wilkinson in September 1995 on the occasion of the sesquicentenary of the foundation of the Chemistry Department at Imperial College. The tapes are now in the National Sound Archive of the British Library, accession nos. H7931-2.

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