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Review

Is the silver–alkene interaction a useful new supramolecular synthon?

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Contents

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ABSTRACT

The use of the silver–alkene interaction in coordination and supramolecular chemistry is surveyed in this review. Although organometallic complexes of ethene date back almost 200 years to the historic example of Zeise's platinum salt, the use of alkenes as donor ligands for silver(I) has a much more recent history. A diverse range of interesting silver–ethene complexes have been reported in recent years, whilst numerous discrete silver(I) complexes of structurally diverse olefinic ligands have been the subject of much study over the last fifty years. More recently, ligands containing multiple alkene subunits have been used for the synthesis of 0-, 1-, 2- and 3D supramolecular assemblies, which is the major focus of this review.

1. Introduction

Metallosupramolecular chemistry [1,2] involves the use of combinations of bridging organic ligands and metal atoms for the construction of both discrete and 1-, 2- or 3D polymeric assemblies with diverse molecular architectures [3–9]. Simply by mixing together the components, self-assembly processes typically lead to high yields of the resulting species in a single step, directed by distance and angular information encoded into the metal and ligand components. We have long been involved in the synthesis and study of both chelating [10] and bridging [11] heterocyclic ligands in both coordination chemistry and metallosupramolecular chemistry. We have been especially interested in ligand design concepts [12].

In recent years silver(I) has proved to be a popular and versatile metal for the formation of supramolecular assemblies [13–17]. The d^{10} silver(I) unit represents a promiscuous species that offers a range of coordination numbers and geometries. For example, a recent analysis [17] of the Cambridge Structural Database found the following percentages of coordination number for silver(I): two-coordinate (24.2%), three-coordinate (22.7%), four-coordinate (43.9%), five-coordinate (4.7%), six-coordinate (3.9%), seven-coordinate (0.3%) and eight-coordinate (0.3%). Most commonly, silver has been employed with bridging heterocyclic ligands, although other functional groups have been used for bonding to this metal. We have recently reviewed the use of silver–pyrazine interactions as a resourceful synthon for the preparation of diverse metallosupramolecular species [18].

In this review we describe the history of silver–alkene interactions and assess the potential of this interaction as a useful synthon in metallosupramolecular chemistry. Within this framework we describe some of our initial results in the use of this interaction to assemble a number of supramolecular assemblies.

2. Discussion

2.1. Silver–ethene complexes

Zeise's salt, $K[PtCl_3(C_2H_4)]$, was the first organometallic compound isolated in pure form and its discovery in 1825 is generally

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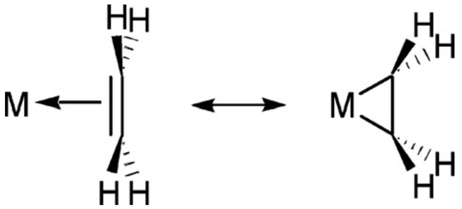


Fig. 1. Resonance contributors to the structure of an ethene–metal complex.

considered to mark the birth of organometallic chemistry [19]. This salt contains a η^2-coordinated ethene molecule which is oriented perpendicular to the coordination plane of the platinum. The bonding of the ethene molecule in this, and the many other transition metal complexes of ethene that have been reported since [20], is explained by the Dewar–Chatt–Duncanson model [21–23]. In this model the ligand donates electron density from its HOMO π orbital to the metal and accepts electron density from the metal into its π^* LUMO (Fig. 1). This back-bonding results in a lengthening of the C=C bond distance and pyramidalisation of the carbon atoms.

The first silver–ethene complexes were prepared by Quinn and Glew in 1962 [24] who reacted solid silver(I) tetrafluoroborate with gaseous ethene to obtain $Ag(C_2H_4)_n{}^+$ salts ($n = 1–3$). Such salts have been the subject of many subsequent studies, both experimental and computational [25–33]. Experimental bond dissociation energies (BDEs) of $Ag^+(C_2H_4)_n$ are 135, 126, 57, 27 and 18 kJ/mol for $n = 1–5$, respectively, with the BDE of the sixth ligand estimated to be 14 kcal/mol [34], values that agree with computational studies [33]. These studies show that the strength of the silver–ethene interaction decreases for the coordination of each subsequent ethene ligand and that species with $n > 3$ are not particularly stable. The extent of back-bonding in these complexes has been the subject of some debate, but appears to be relatively weak. The bonding of ethene to silver is weaker than to gold [33] and in such complexes ethene is readily displaced by other ligands (including solvents). Nevertheless, the interaction is still sufficiently strong for potential use in supramolecular synthesis [35]. Related complexes of ethene with small silver clusters have been the subject of a number of recent studies [34,36–38].

Over the last decade the structures of a number of stable silver–ethene complexes have been determined by single crystal X-ray crystallography [39–47]. These involve the use of highly fluorinated tris-pyrazolylborates [39–42] or diazapentadienyl ligands [44] to stabilize the ethene coordination. The scorpionate complexes prepared by Dias and co-workers have also been the subject of computational studies [48] and are useful catalysts for carbene insertion into hydrocarbons [49]. Fig. 2 shows the structure of the complex with tris(3-trifluoromethylpyrazolyl)methylborate (**1**), which features a tetrahedral silver atom bound to three scorpionate nitrogen atoms and a η^2-ethene. Reisinger et al. [45] have reported the structure of the first stable tris–ethene adduct, preparted as a salt with the $[Al\{OC(CF_3)_3\}_4]^-$ anion (Fig. 3). Interestingly, the three ethene ligands all lie in the coordination plane, rather than perpendicular to it. An intriguing trinuclear cage-like complex (Fig. 4) was reported by Krossing et al. [46] which consists of three $Ag(C_2H_4)$ units capped by two $^-OC(CF_3)_3$ trifurcated bridging anions. This area has been the subject of an excellent recent review [43].

2.2. Discrete silver–alkene complexes

Silver(I)–alkene complexes have been known for over seventy years, ever since Winstein and Lucas's pioneering study on the interaction of silver(I) ions with a range of unsaturated compounds [50]. Throughout the 1950s and 1960s various groups reported studies of a number of discrete silver–alkene complexes that were characterised in both solution and the solid state [51–54]. IR studies usually indicate the coordination of olefins to silver(I) in the solid state by a shift in the C=C stretching vibration to lower frequencies compared to the uncoordinated olefin [55]. ^1H NMR spectra of coordinated alkenes show a characteristic downfield chemical shift compared to the parent alkene, the magnitude of which is dependent upon the nature and substitution of the double bond [56].

The use of the silver(I)–alkene interactions for the separation of unsaturated hydrocarbons is an established practice with silver(I) nitrate a commonly used reagent. Methods of separation include impregnating silver(I) nitrate upon solid supports such as silica gel, alumina, amberlyst resin and Sephadex, HPLC and precipitating solid silver(I) complexes with the target compound out of a solution mixture. These methods can be applied to the separation of geometric isomers and constitutional isomers that differ in the placement of olefinic groups, the separation of alkene-containing compounds of different chemical formulae and alkene impurities

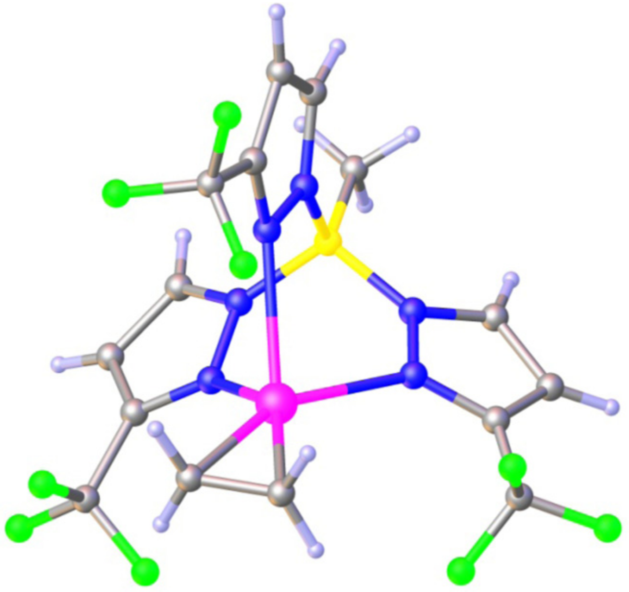


Fig. 2. A discrete ethene complex with a scorpionate ligand [42]. In all figures the following colour coding is employed: silver – large pink; carbon – grey; hydrogen – small light blue; nitrogen – dark blue; oxygen – red; boron – yellow; fluorine – light green; sulfur – yellow; chlorine – dark green.

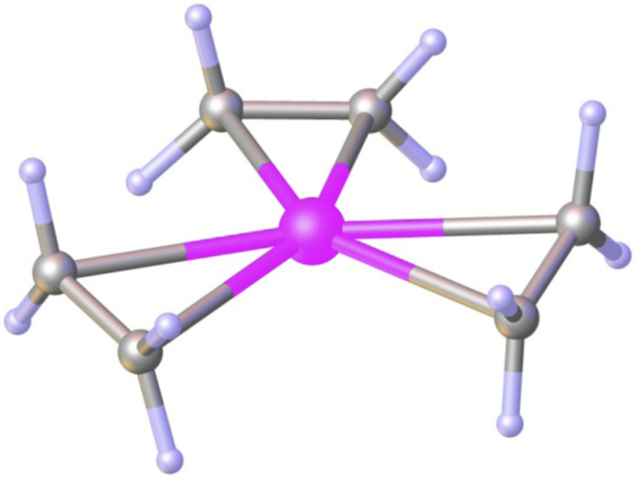


Fig. 3. The cation in the first tris–ethene–silver complex [45].

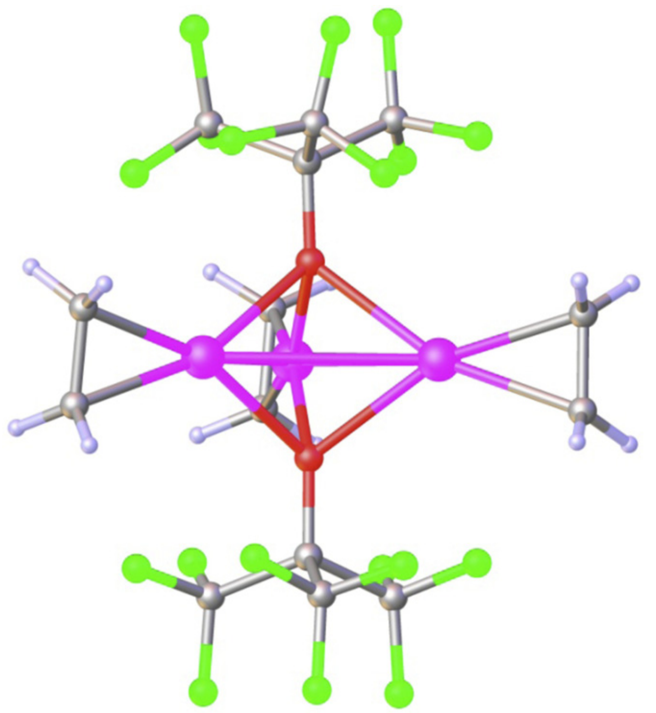
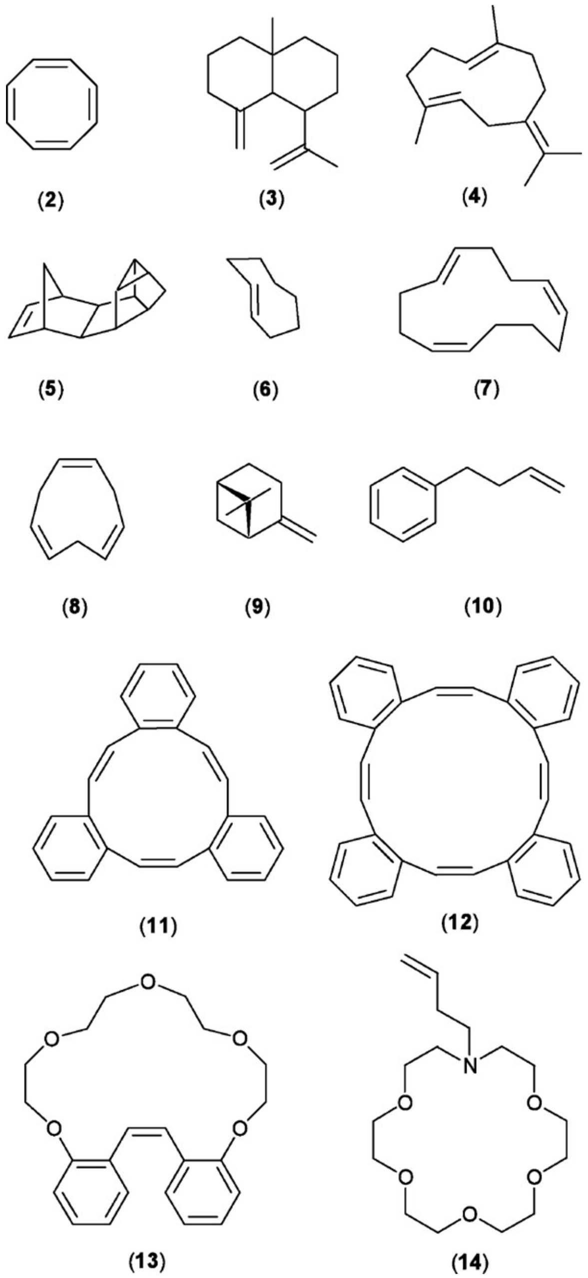


Fig. 4. A cationic trinuclear silver–ethene cage [46].

from alkanes. Such applications have been the subject of reviews [57,58].

The first X-ray crystal structure of a silver–alkene complex was that formed from reaction of silver nitrate with cyclooctatetraene (**2**) (Scheme 1), in which the ligand acts in a chelating mode with coordination of two distal C=C bonds (Fig. 5), albeit with stronger bonding to one of the alkene units [59]. The formation of crystalline silver adducts of non-crystalline alkenes has been used to determine the structures of olefinic hydrocarbons. These include natural products, such as β-gorgonene (**3**) [60] and germacratriene (**4**) [61], and strained or unusual hydrocarbons, such as 4,7-methano-2,3,8-methenocyclopent[α]indene (**5**) [62], *trans*-cyclooctene (**6**) [63], *cis*- and *trans*-cyclodecene [64,65], 1,5-cyclooctadiene [66], *cis*,*trans*,*cis*-1,5,9-cyclotridecatriene (**7**) [67] and *cis*,*cis*,*cis*-1,4,7-cyclononatriene (**8**) [68]. Interestingly, in the complex with (**7**) a single silver atom coordinates to all three C=C double bonds, whilst ligand (**8**) forms a trinuclear adduct (Fig. 6). We have also reported [69] the preparations and X-ray structures of a number of discrete silver complexes of chiral monoterpenes, such as (−)-β-pinene (**9**) (Fig. 7).

More recently, the X-ray structure of a discrete silver(I) complex of 4-phenylbut-1-ene (**10**) has been reported, in which the ligand acts in a chelating mode in its silver tetrafluoroborate complex (Fig. 8), being bound in a η^2-fashion to the alkene unit and a η^1-mode to the phenyl ring [70]. Curiously, this was described as “the first chelating olefin” despite the fact (*vide supra*) that the very first crystal structure of a silver–alkene complex involved a chelating diene. In contrast, styrene has been recently reported to form a discrete mononuclear complex, within which the vinyl group coordinates in preference to the benzene ring [71]. In the context of the present review, this is an interesting result as silver–arene interactions have a more established history in metallosupramolecular chemistry, most notably through the excellent work of Munakata et al. [72]. In a similar way, the two annulenes (**11**) and (**12**) each react with silver salts to form discrete adducts (Fig. 9), within which the silver coordinates to the C=C double bonds in preference to the benzene rings [73,74].



Scheme 1.

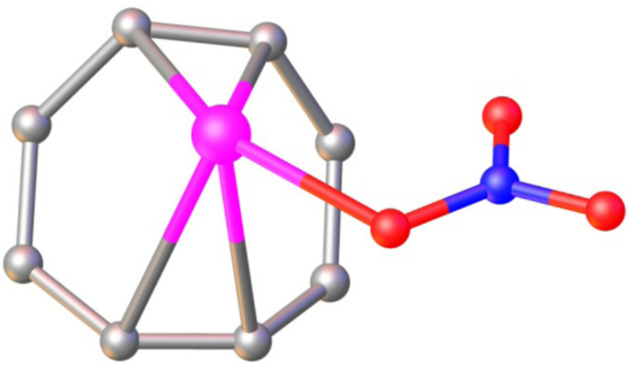


Fig. 5. The first crystallographically characterised silver alkene complex [59].

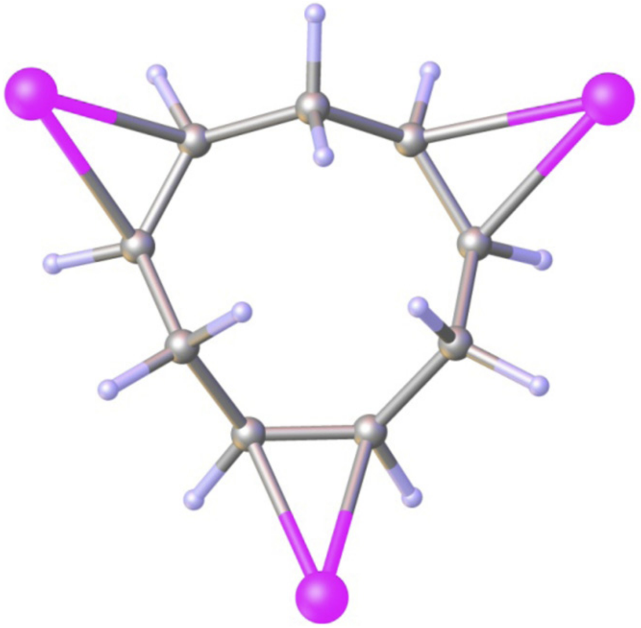


Fig. 6. A trinuclear complex derived from ligand **8** [68].

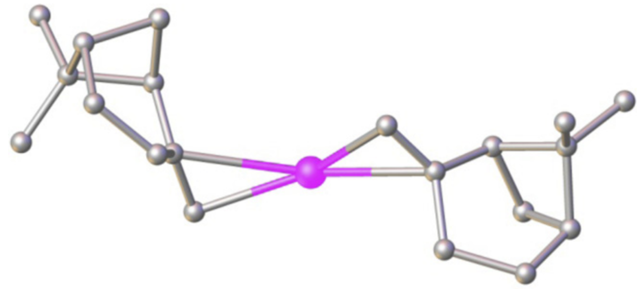


Fig. 7. A discrete silver complex of the chiral monoterpene (−)-β-pinene (**9**) [69]. Hydrogen atoms omitted for clarity.

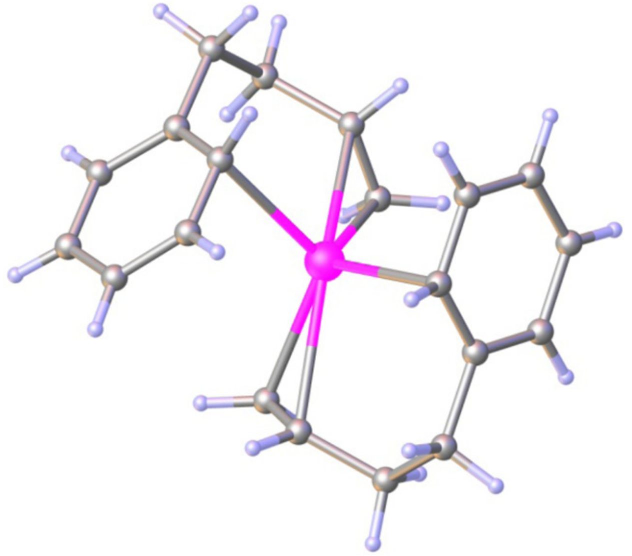


Fig. 8. The discrete complex derived from ligand (**10**) [70].

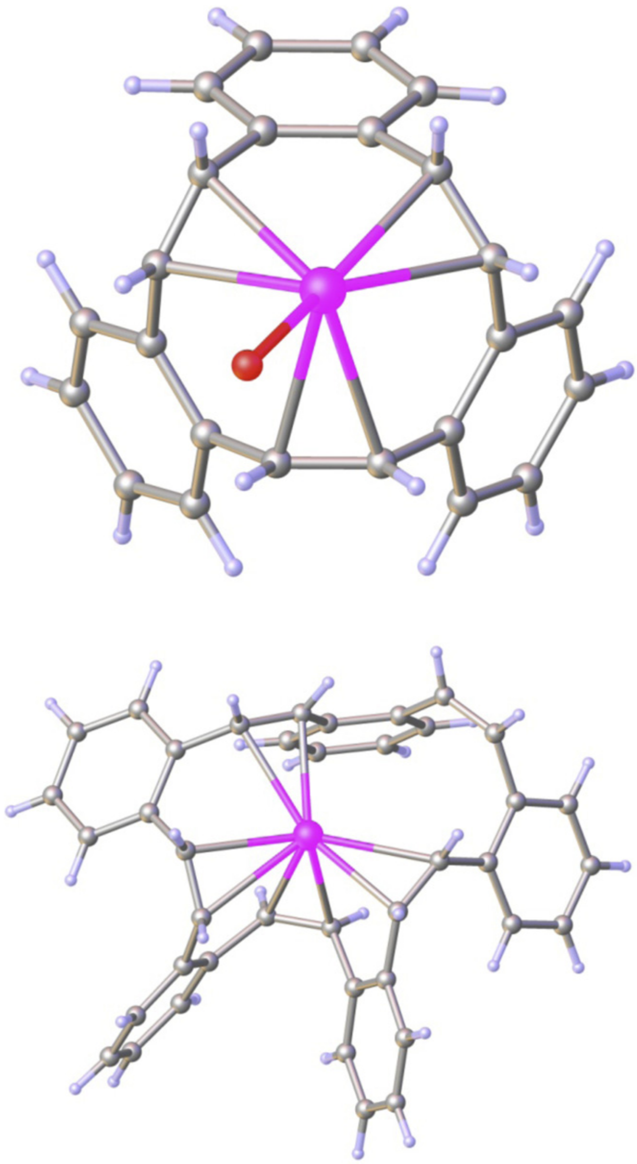


Fig. 9. The structures of the silver complexes of annulenes (**11**) and (**12**) [73,74]. In the complex of (**11**) one oxygen of the coordinated triflate counteranion is shown.

Coordinating alkene subunits have also been incorporated into the structures of crown ethers. For example, the crown ether (**13**) forms a discrete complex with silver(I) wherein the silver binds weakly to three of the ether oxygen atoms but more strongly to the C═C double bond [75]. Similarly, the lariat ether (**14**) forms a discrete silver complex (Fig. 10) within which the ether encapsulated silver atom is also bonded to the pendant butenyl arm of the crown ether [76].

On the basis of the above reports it is evident that the silver–alkene interaction is a robust one; the potential of this as a supramolecular synthon will be discussed in the next section.

2.3. Supramolecular assemblies

In the crystal packing of some of the discrete structures discussed in the previous section the molecules assemble into higher aggregates through weak intermolecular interactions often involving bridging by weakly coordinated anions. In this section, we expand this concept to the use of ligands containing multiple alkene

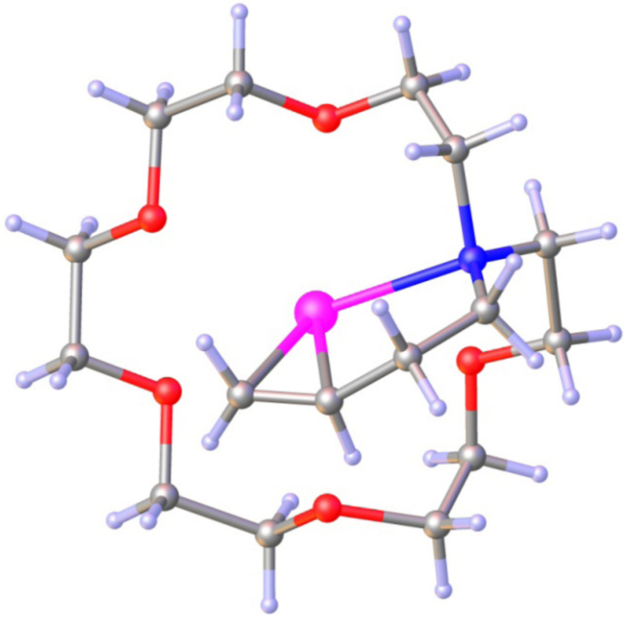
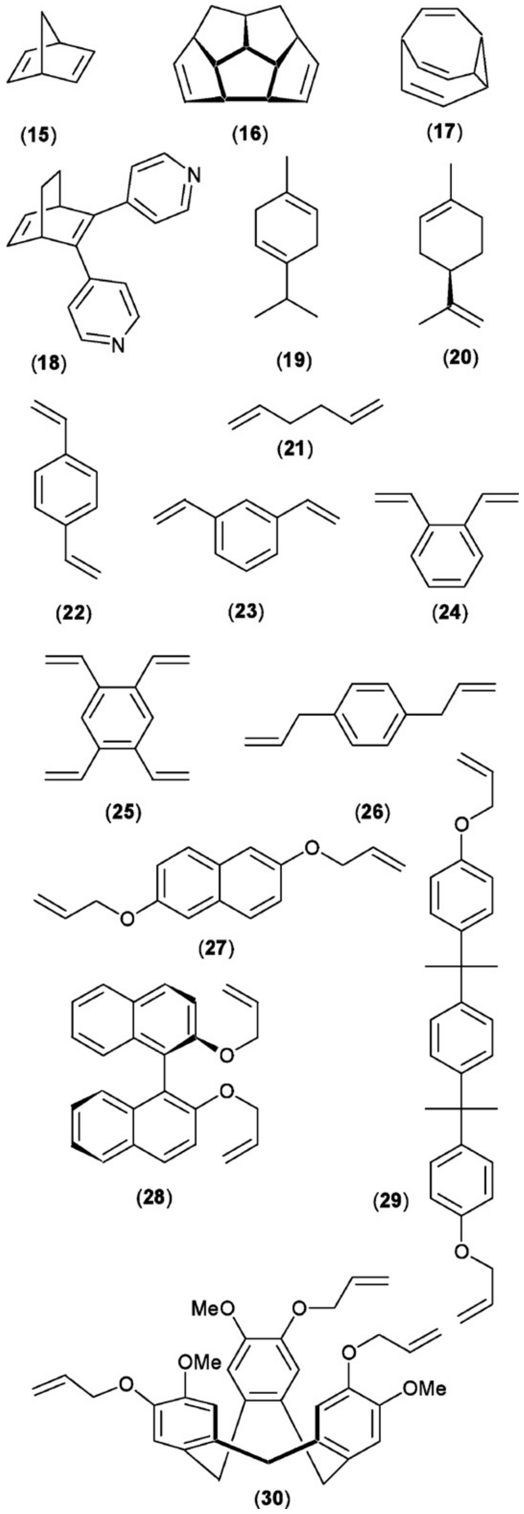


Fig. 10. Silver complex of the lariat crown ether (**14**) [76].

subunits as bridging ligands for the rational construction of 1-, 2- or 3D supramolecular assemblies.

A number of early studies of silver alkene complexes involved various types of supramolecular species. For example, silver nitrate was long known to form both 2:1 and 1:1 complexes with norbornadiene (**15**) (Scheme 2), which were postulated to be polymeric assemblies [54]. The X-ray crystal structure of the 2:1 species was reported as early as 1966 [77], corrected in 1983 [78] and determined to higher precision in 2006 [79]. This compound is a 1D coordination polymer (Fig. 11), with a ladder-like structure within which the diene (**15**) bridges two silver atoms providing the rungs of the ladder with the bidentate nitrate anions providing the sides of the ladder. The structure of the 1:1 species was finally [79] revealed to be a 2D assembly involving silver atoms bridged by both the diene and anions in an extended 2D array. The corresponding 1:1 complex with silver perchlorate has a very similar 2D network [80]. Other bridged polycyclic hydrocarbons generate similar species with silver salts. Examples include substituted norbornadienes [81], the pentacyclic diene (**16**) [82] and bullvalene (**17**) (Fig. 12) [83], the latter of which had previously been reported to form a discrete complex with silver tetrafluoroborate with a silver atom bound to three bullvalene molecules [84].

Our own involvement in this field arose from attempts [85] to rationally assemble metallosupramolecular triangles using ligands such as the bicyclooctadiene (**18**), which we hoped would provide the requisite 60° angle for triangle formation. In the event, reaction of (**18**) with silver(I) nitrate furnished a product wherein the silver atom not only coordinates to both pyridine nitrogen atoms but also to the more exposed double bond in the bicyclooctadiene unit (Fig. 13). We have since begun a systematic study of the use of bridging ligands which possess multiple alkene subunits as synthons for supramolecular assembly. As an extension of our work with monoterpenoid alkenes as ligands, we showed that both γ-terpinene (**19**) and (+)-limonene (**20**) could be used to generate coordination polymers upon reaction with silver triflate and silver hexafluorophosphate, respectively [69]. The latter of these is a chiral coordination polymer (Fig. 14), which themselves are the subject of much current interest [86,87].



Scheme 2.

We next turned our attention to the use of simple hydrocarbons containing multiple terminal alkene (vinyl and allyl) units as potential bridging ligands. There had been a few isolated examples of such hydrocarbons being used to prepare silver-based assemblies. For example, in an early report 1,5-hexadiene (**21**) was reported to react with silver perchlorate to give a discrete binuclear species (Fig. 15), wherein (**21**) acts as both a chelating and bridging ligand [88]. We prepared the three isomeric divinylbenzenes (**22**–**24**) and examined their reactions with various silver(I) salts, which generated both discrete and polymeric assemblies [89]. The *para*-isomer (**22**) reacted with silver perchlorate to give a discrete M_2L_2

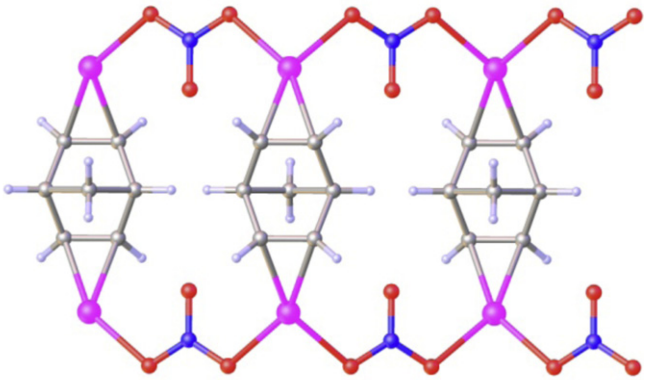


Fig. 11. The X-ray structure of the much studied 2:1 silver–norbornadiene ladder-like assembly [77–79].

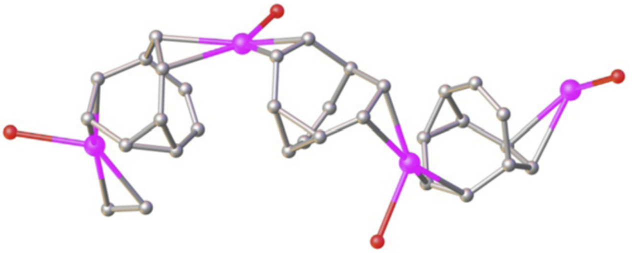


Fig. 12. The polymeric assembly formed by bullvalene (**17**) [83]. The third ligand bound to silver is a water molecule. Hydrogen atoms not shown.

dimetallocycle (Fig. 16), whereas the *meta*-isomer (**23**) produced a discrete M_2L_3 cage-like assembly with silver tetrafluoroborate, but 1D coordination polymers with silver perchlorate or triflate (Fig. 17). Interestingly, the *ortho*-isomer (**24**) reacted with silver perchlorate to give a species (Fig. 18) within which three silver atoms bind in a η^2-fashion to two vinyl groups and the benzene ring, all on the same face of the ligand, as part of a complex 2D extended structure. Such combined alkene/arene coordination has been observed previously in silver complexes of arylpolyenes [90,91].

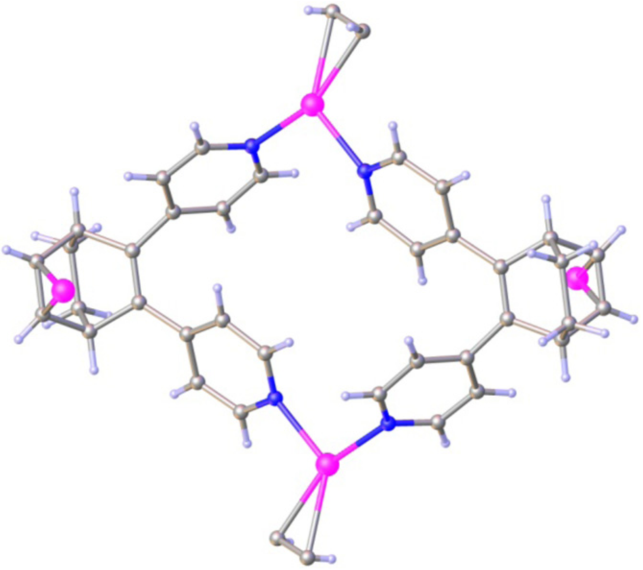


Fig. 13. A section of the extended polymeric structure of the silver nitrate complex of ligand (**18**) [85]. Only the alkene carbons of adjacent ligands are shown.

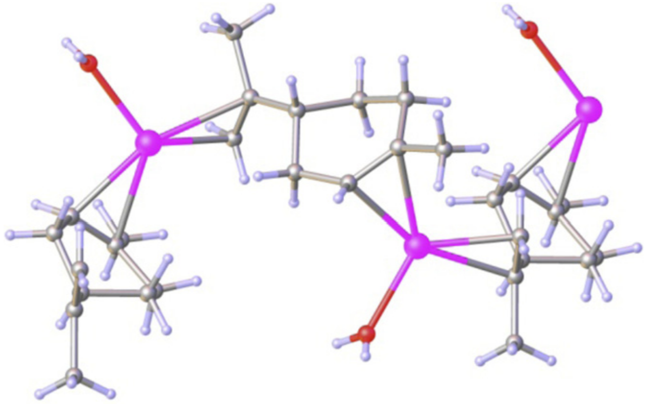


Fig. 14. A section of the 1D chiral coordination polymer derived from the monoterpene (+)-limonene [69].

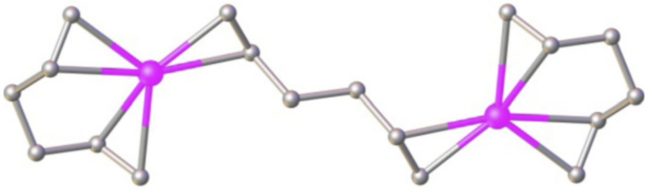


Fig. 15. The binuclear silver complex of 1,5-hexadiene [88]. Hydrogen atoms not shown.

We have since extended this concept to greater numbers of vinyl groups. For example, 1,2,4,5-tetravinylbenzene (**25**) reacts in a variety of ways with silver salts [92]. Reaction with silver tetrafluoroborate results in the formation of an interesting silver sandwich (Fig. 19), wherein two ligands gather together four silver atoms into a planar array. This assembly is reminiscent of the hexanuclear silver platter that we previously reported using a hexapyridyl[3]radialene ligand [93].

Such assemblies are not restricted to vinylbenzenes, but are equally accessible using allylbenzenes [92]. For example, 1,4-diallylbenzene (**26**) forms a linear 1D coordination polymer upon reaction with silver perchlorate (Fig. 20). The incorporation of the extra methylene groups adds additional flexibility to the arms of the ligand and, of course, increases the separation between the silver atoms in the polymeric assembly to a distance of 11.93 Å, compared to a distance of 8.04 Å for the divinyl analogue. These polymeric chains pack together through intimate π–π stacking interactions of the benzene rings.

We have also introduced oxygen atoms into the ligand arms and incorporated central cores other than benzene. Thus, we have prepared variously substituted diallyloxy-benzenes and -naphthalenes, which all act as bridging ligands to produce interesting assemblies upon reaction with silver salts [92]. For example, 2,6-diallyloxynaphthalene (**27**), readily prepared by reaction of

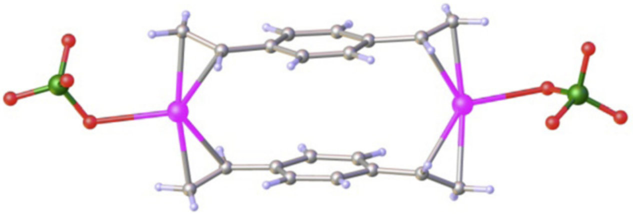


Fig. 16. The dimetallomacrocycle formed from ligand (**22**) [89].

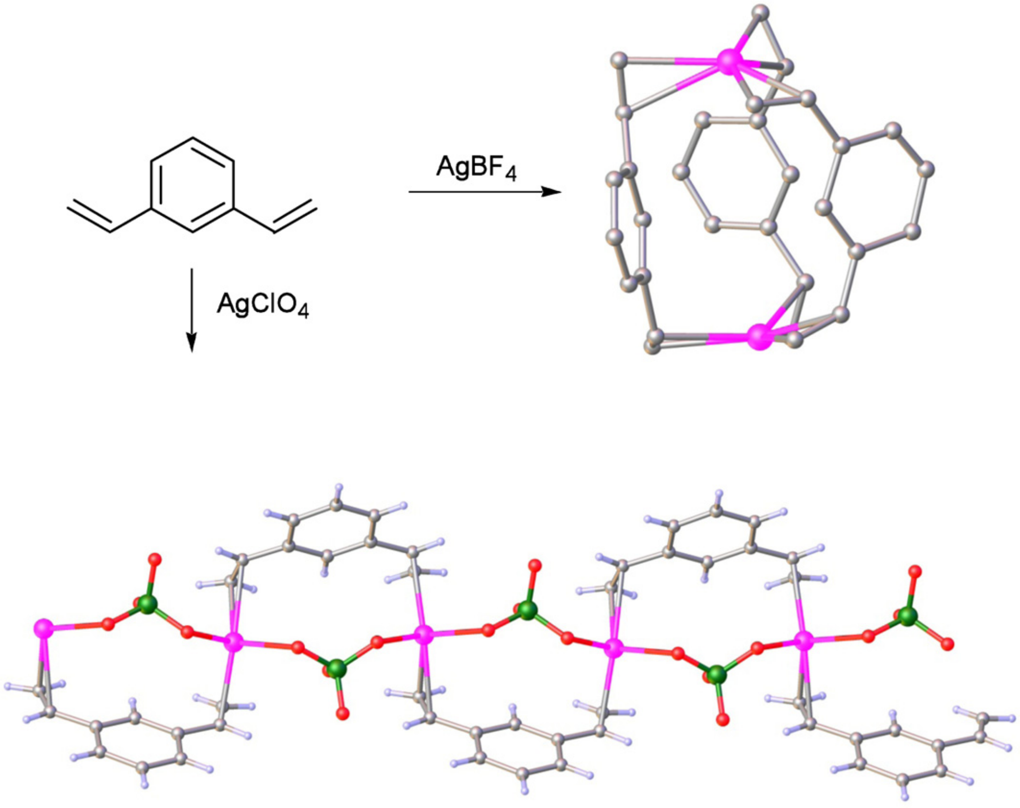


Fig. 17. Products produced by reaction of (**23**) with $AgBF_4$ and $AgClO_4$ [89].

2,6-dihydroxynaphthalene with allyl bromide, reacts with silver tetrafluoroborate to produce a complicated 2D polymeric assembly. Within this structure there are two crystallographically independent ligands, one of which acts as a two connecting unit via the two alkene units, whilst the second acts as a four connector using the two alkene arms and double η^2-coordination of the naphthalene ring (Fig. 21). The silver atom acts as a three connecting unit, being bound to two alkene units and a naphthalene ring. Naturally, the extra atoms in the arms and the greater size of the naphthalene core serve to increase the separation between the metal atoms to 13.5 Å. Interestingly, in this and related structures the silver atoms prefer to coordinate to the alkene units, and in some cases the naphthalene rings, in preference to the ether oxygen atoms.

Chirality can also be introduced into ligands of this sort and incorporated into the resulting supramolecular arrays. For example, reaction of 2,2′-dihydroxy-1,1′-binaphthyl (BINOL) with allyl bromide produces ligand (**28**), which upon reaction with silver salts leads to chiral assemblies by virtue of the atropisomeric nature of

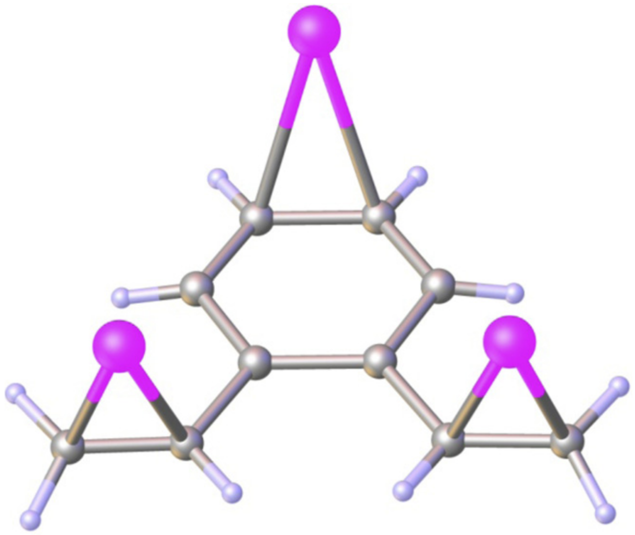


Fig. 18. The coordination of ligand (**24**) to three silver centres [89].

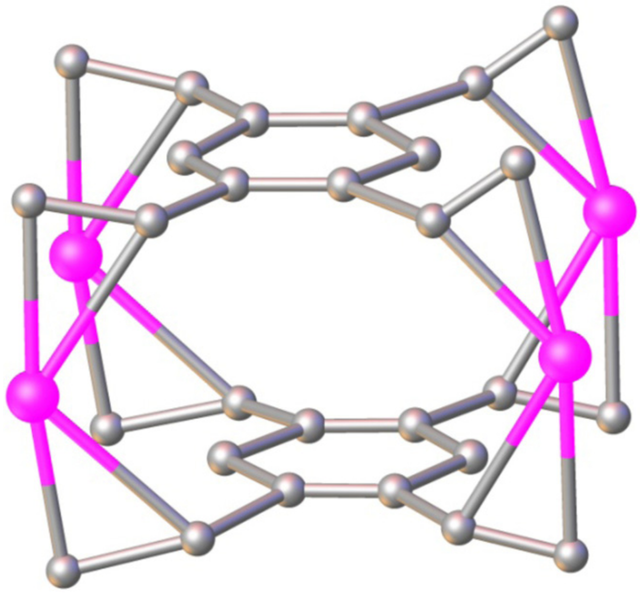


Fig. 19. The silver sandwich formed from ligand (**25**) [92]. Hydrogen atoms not shown.

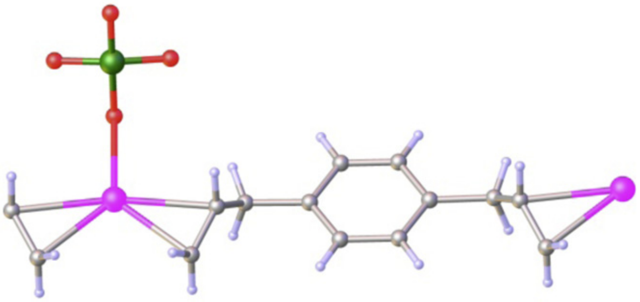


Fig. 20. The 1D polymer formed by *para*-diallylbenzene [92].

the ligand. We have synthesised these complexes in both racemic and homochiral forms [92]. The X-ray structure of the product from reaction of the racemic ligand with silver perchlorate is shown in Fig. 22. This is a discrete M_2L_2 binuclear complex within which the ligand acts in a doubly chelating mode with each silver atom being coordinated to an alkene unit and having η^2-coordination to the distal naphthalene ring. The silver atoms are also coordinated to a perchlorate counterion and an acetone molecule from the solvent. An interesting aspect of this reaction is the way in which coordination to silver results in a scissor-like response from the ligand. Fig. 23 shows a comparison of the torsional angles, viewed down the transannular bond, in the X-ray structures of the free ligand and the complex, wherein the torsional angle between the naphthyl rings changes from 69° in the free ligand to 110° in the binuclear complex.

Much greater separation between the metal centres can be achieved using ligands derived from precursors having two well separated phenol units. Recently, we reported the syntheses of a family of bridging N-heterocyclic ligands derived from commercially available bis-phenols [94]. As an extension of this design strategy we prepared ligand (**29**) from reaction of bis-phenol P with allyl bromide [95]. Upon reaction with silver tetrafluoroborate this ligand furnishes a 1D zig–zag coordination polymer (Fig. 24) within which the silver atoms are separated by a distance in excess of 2 nm.

In related recent work, Hardie and co-workers extended their interesting work on cyclotriveratrylene-based ligands [96] to the

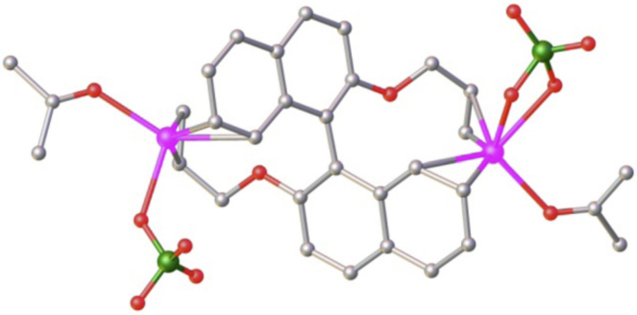


Fig. 22. The discrete M_2L_2 species formed from ligand (**28**) [92]. Hydrogen atoms not shown.

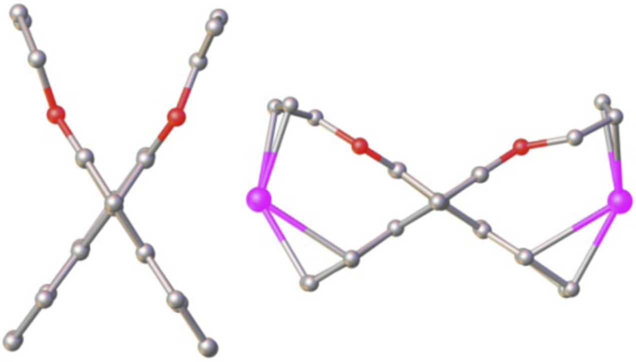


Fig. 23. Comparison of the dihedral angles between the naphthalene rings in the free ligand and silver complex of (**28**) [92]. Hydrogen atoms omitted for clarity.

tris-allyl derivative (**30**), which produced an intriguing array of complex network structures and one discrete complex upon reaction with various silver salts [97]. In all of these, some or all of the allyl arms displayed η^2-coordination to the silver atoms, which in some cases were also coordinated to aryl and/or ether oxygen groups. Depending on reaction conditions, $AgSbF_6$ gave 3D coordination polymers with unusual topologies or a discrete M_2L_2

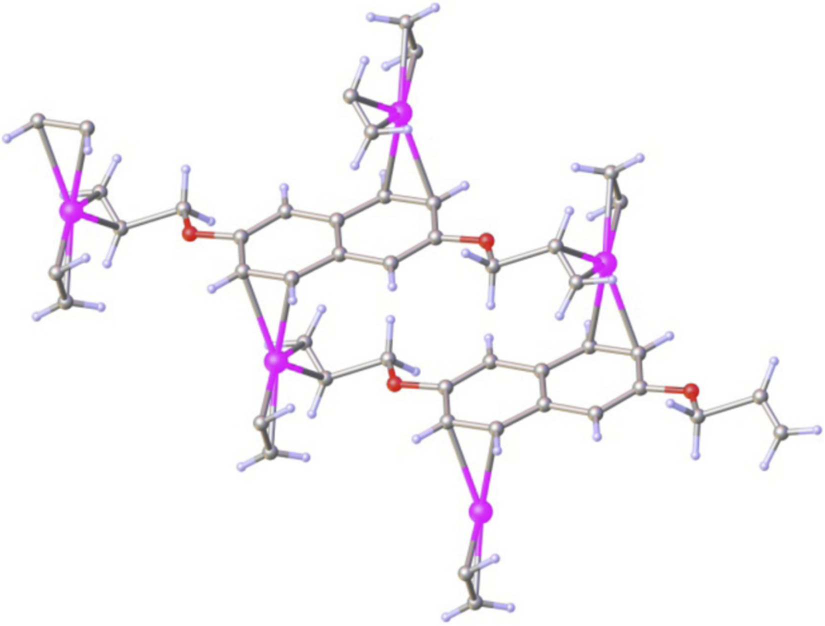


Fig. 21. A section of the 2D coordination polymer obtained from ligand (**27**) [92]. Only the directly bound carbons of adjacent ligands are shown.

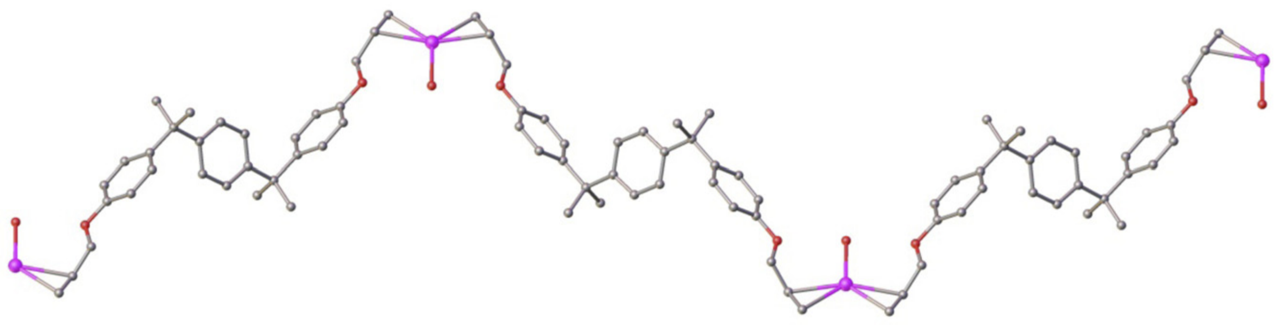
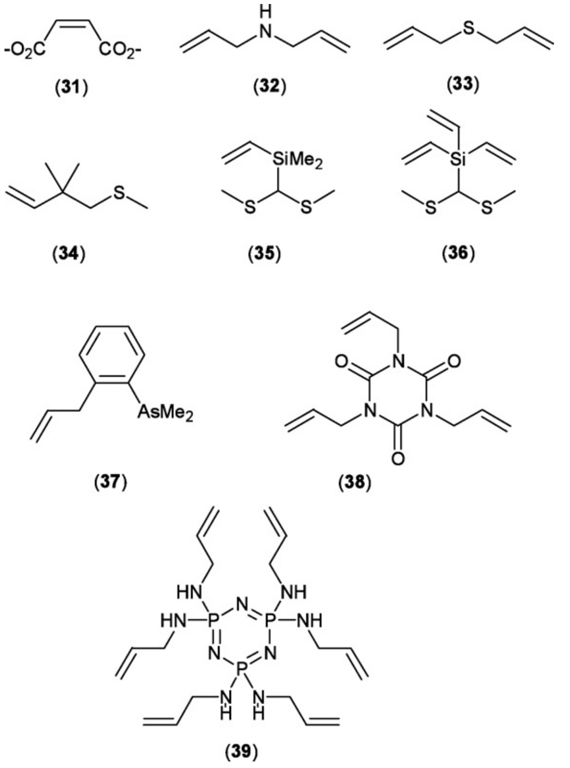


Fig. 24. A section of the 1D polymeric assembly produced by ligand (**29**) [95]. The third ligand bound to silver is a water molecule. Hydrogen atoms not shown.



Scheme 3.

capsule. Silver tetrafluoroborate and perchlorate gave isostructural chain polymers, whilst silver triflate gave 2D and 3D coordination polymers with bridging triflate anions.

Several other ligands have been reported that contain alkene subunits that act as bridges in conjunction with other donor groups within the same ligand. For example, whereas the N-butenyl lariat ether (**14**) forms a discrete chelating complex (*vide supra*), the corresponding N-allyl ligand acts as a bridging ligand with silver to form a 1D coordination polymer [98]. Other examples of bridging include the ligands shown in Scheme 3, all of which have been used to bridge silver atoms in crystallographically characterised assemblies. The simple maleate dianion (**31**) has been reported to use all its oxygen and alkene donors to bridge five silver atoms [99]. Both diallylamine (**32**) and diallylsulfide (**33**) chelate to one silver atom using the two allyl arms and bridge to a second silver via the nitrogen/sulfur atom (Fig. 25) [100,101]. The sulfide (**34**) reacts with silver trifluoroacetate to chelate through the vinyl and sulfur atoms with further bridging of the sulfur to another silver [102]. The vinylsilyldithioactals (**35**) and (**36**) form polymeric assemblies involving chelation by the vinyl and sulfide groups [103]. In an early study, ligand (**37**) bridged two silver atoms via the allyl and arsine donors as part of a polymeric assembly involving bridging nitrate counterions [104]. Very recently, the triallyl triazine (**38**) was reported to form a 1D coordination polymer involving bridging by two of the three allyl arms [105]. Finally, the cyclotriphosphazene (**39)** with six appended allyl arms formed an intriguing 1D coordination polymer involving coordination by all six arms [106]. Interestingly, this was then isomerised into a 3D polymer. This design concept was further extended to a twelve armed ligand that the authors described as a "silver ion sponge", due to its ability to use all twelve allyl arms to gather together five silver atoms [106].

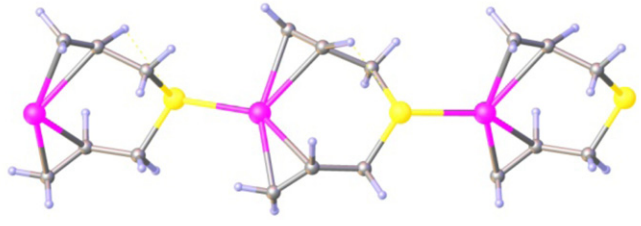


Fig. 25. The 1D coordination polymer formed by diallylsulfide (**33**) [101].

3. Conclusion

In this review we have surveyed the history and use of the silver–alkene interaction in the context of its potential use as a useful new supramolecular synthon. As described above, several interesting silver–ethene complexes have been characterised in recent years. Numerous other discrete complexes have been reported which have resulted from the reactions of silver(I) salts with a diverse array of olefinic compounds. This has led to the rational use of the silver–alkene interaction for the assembly of diverse 0-, 1-, 2- and 3D supramolecular assemblies using readily synthesised bridging ligands containing multiple alkene subunits. We contend that, like other cation–π interactions [107], this supramolecular synthon has enormous potential for the development of a diverse range of metallosupramolecular species.

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