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Coordination Chemistry Reviews
204 (2000) 113–171

COORDINATION CHEMISTRY REVIEWS

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Poly- and oligometalloporphyrins associated through coordination

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Received 28 April 1999; accepted 21 July 1999

Contents

Abstract

Significant developments in the construction of oligoporphyrins and oligometalloporphyrins supply a large variety of new interesting compounds with potential use in electron and energy transfer. Their physicochemical properties depend primarily on the nature of the linkage between porphyrin building blocks. In this review we have focused on the expanding role of the coordination bond in creation of polyporphyrin architecture.

Keywords: Porphyrin; Metalloporphyrin; Oligomers; Self-assembly

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0010-8545/00/$ - see front matter
PII: S0010-8545(99)00207-6

1. Introduction

The design of discrete ordered arrays of porphyrins and metalloporphyrins has attracted much interest recently, and produced a synthetic methodology with a building block approach. Generally, multiporphyrin systems have been considered as biomimetic models or materials for the transport of energy, charge, molecules and ions and as potent catalysts. Thus these arrays are useful whenever a specific arrangement of porphyrin/metalloporphyrin moieties in space is crucial for an efficient operation.

Porphyrin assemblies are of fundamental importance as models for the study of the energy and electron transfer in the light-harvesting antenna and the photosynthetic reaction centers [1–6]. Particularly, the discovery of the special pair in Photosystem I motivated the search for the relevant model diporphyrin arrangements. This special pair consists of two bacteriochlorophyll monomers, which are roughly coplanar and demonstrate a high degree of macrocyclic overlap with a separation of approximately 3.6 Å [7–9]. Consequently, investigations have been designed to mimic the structure and/or function of the photosynthetic reaction center, including the primary events of the photosynthetic process that result in charge separation and successive electron transfers. Simultaneously, synthetic procedures have been developed to obtain multiporphyrin arrays that can be important for the solar energy conversion and storage [10,11]. Such investigations may eventually lead to use the light energy absorbed by oligoporphyrins (oligometalloporphyrins) to drive endothermic reactions, such as splitting of water to H_2 and O_2 which can be used as a fuel source [12].

Many examples of biochemical reactions are found in enzymatic systems (including hemoproteins) that have more than one metal ion in the active center. For instance, several aspects of the cooperative binding of dioxygen by hemoglobin, which contains four hemes, have been explored using oligomeric metalloporphyrins [13–15]. Cytochrome *c* oxidase carries out a four-electron reduction of dioxygen to water using two heme and two copper centers. Accordingly, oligomeric metalloporphyrins have been applied with success to investigate mechanistic aspects of this process [16–20]. The decomposition of H_2O_2 by metallodiporphyrins has been also investigated in light of catalase reactivity [21,22]. An action of a tetranuclear manganese cluster of Photosystem II has been reproduced by manganese porphyrin dimers which contributed to an electrocatalytic evolution of dioxygen from water [23]. Finally, some features of dinitrogen binding by nitrogenase have been explored in the oligometalloporphyrin environment [19,24,25]. A cooperative action of the metal ions of metallodiporphyrins (or oligomeric metalloporphyrins) in activation of the small molecules such as H_2 [19,26] or CO [27] has been observed as well. The structurally rigid polymeric metalloporphyrins may act as a fragment of the catalytic surface where the substrates bound to the metallic centers are activated to undergo the required chemical transformation. In a sense these arrays can be treated as the perspective polycentric homogenous catalysts [28,29].

Oligomeric porphyrins and metalloporphyrins with various structures are also under intensive investigation due to the relevance to their potential application in

molecular electronics and as new photonic materials [4,10,30–42]. For instance, the multiporphyrin tapes or arrays may serve as molecular wires [30–33,42], molecular switches [34,40,41], photon funnels [10], and molecular elements of information storage [37]. The construction of extended π-systems resulted in properties required for third-order nonlinear optical materials [41]. Multispin molecular assemblies featuring metalloporphyrins are promising components of new magnetic materials [43–46]. Interestingly, some oligomeric porphyrins have been tested for photodynamic therapy of tumors [47,48].

Another field of research on porphyrin oligomers has focused on selective molecular recognition that can result from the specific geometry of cavities surrounded by porphyrin planes [48–52]. In the extreme case, i.e. in the crystal engineering of porphyrins and metalloporphyrins, nanoporous molecular crystalline solids have been constructed [53]. Such architecture with large channels, formed from catalytic active metalloporphyrins, may be useful for designing shape selective oxidation catalysts [53].

Porphyrins and metalloporphyrins provide an advantageous class of building blocks for the construction of large multicomponent architecture because of their relatively facile synthesis, stability, and diversity of their properties. The controlled modifications of monomeric porphyrins and metalloporphyrins to create required structural and electronic properties have emerged as an important research topic [54]. One can suitably determine the overall features of porphyrin including the electronic structure, geometry and an access to the porphyrin (metalloporphyrin) core by means of well-planned peripheral substitutions either at β-pyrrole or *meso*-position(s). In addition to peripheral modifications, core alteration may be effected by replacing the pyrrolic nitrogen atom(s) with other heteroatoms [55], by introducing of one or more additional pyrrole moieties into the macrocycle (expanded porphyrins) [56,57], or by rearranging the methane bridges (porphycene, hemiporphycene, corphycene) [58,59]. Finally, the porphyrins may be modified by inversion of one of the pyrrole rings (inverted porphyrin) [60,61]. These independent routes enable fine tuning of the chemical properties of the monomeric building units.

In order to take an advantage of the abundant library of perspective porphyrin or metalloporphyrin building blocks, the desired peripheral functionality with designed orientation of the binding element(s) is expected to be introduced. Originally, in order to fix chromophores in a certain stereochemical environments, porphyrins were connected by a large variety of covalently bound spacers to avoid ambiguity arising from conformational mobility. More recently an alternative, i.e. a supramolecular approach, has been dynamically explored to create a variety of discrete porphyrin assemblies. The appropriately selected peripheral functionality originates intermolecular interactions of self-assembly. Thus self-assembly makes use of the molecular recognition through hydrogen bond, electrostatic interaction, or coordination of metal ions.

Certain selected aspects of oligoporphyrin and metallooligoporphyrin chemistry were covered in previous review articles [1,6,12,19,62–70]. The primary purpose of this contribution is to look at the examples of oligometalloporphyrin systems in

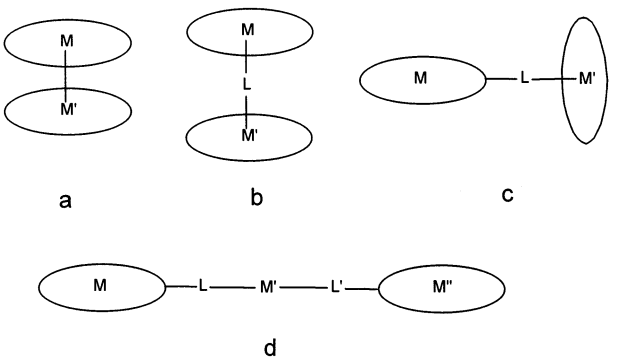
which the porphyrin components are associated by a coordinate metal–ligand bond. Considering a methodology based upon coordination chemistry, oligomers and polymers of metalloporphyrinic building blocks can be rationally designed through selection of the coordination geometry of metals and the structure of the polydentate ligands involved in the supramolecular synthesis. Oligomeric porphyrin and metalloporphyrin assemblies can be stabilized by four structurally different motifs i.e.:

(a) metal–metal bond,
(b) multidentate bridging,
(c) coordination of hybrid metalloporphyrins bearing an additional donor atom at the porphyrin periphery,
(d) bridging by the external metal centers (Scheme 1).

This article concentrates primarily on the architecture of those metalloporphyrins which do form oligomers using built-in external coordination functionality (cases (c) and (d)). However, for the sake of comparison, selected examples of innovative multiporphyrinic arrays built upon other forms of association, including covalent bonding, are presented as well. To give the reader a general overview of these multiporphyrin assemblies, perspective views of essential structures are presented, in which the double bonds within the porphyrins themselves are omitted for clarity.

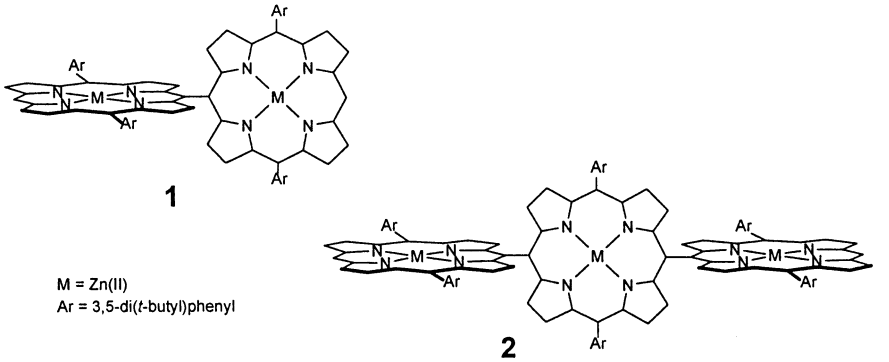
2. Selected examples of multiporphyrin systems linked by covalent bonds

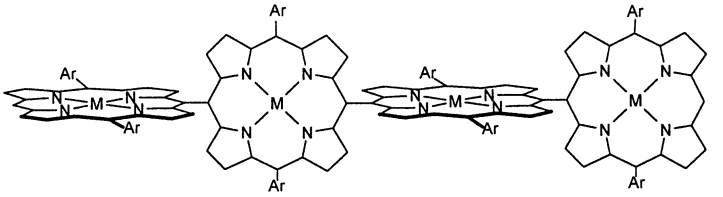
In search for the porphyrin (metalloporphyrin) arrays covalent bonds have been used as the primary means of linking specific building blocks into a single molecule. The nature, geometry, length, and location of covalent bridge(s) offers methods of fine tuning of several properties of these arrays. Here we present selected examples which might provide an overview of the architecture that is already available and



Scheme 1.

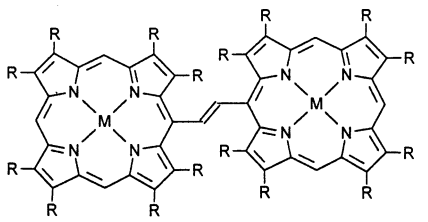
offer some inspiration for the design of the novel systems based upon different modes of binding. Generally, distance, geometry and orientation have been recognized as important factors in control of the efficiency of energy and electron transfer processes. Therefore, oligomeric porphyrin assemblies, in which π-systems can directly interact, are of particular interest. Such an approach resulted in oligomeric porphyrins in which the macrocycles are directly linked through their *meso*-carbons without any spacer [71–79]. Complexes of this kind (for instance, **1**, **2**, and **3**) have been recently synthesized via oxidative coupling of zinc porphyrins where up to eight porphyrinic moieties have been linked into linear arrangements [72,75]. Previously, coupling of oxophlorin π-radicals resulted in formation of non-aromatic dimers with sp^3 hybridization of linked *meso* carbon atoms [80–83]. The stepwise process, where the bridging *meso–meso* fragment was synthesized before the macrocycle closure, has been recently reported [73].





Alternatively, the direct covalent binding of two porphyrin skeletons may involve one *meso*-carbon and β-carbon derived from two different macrocyclic fragments [2,62,84,85], or β positions of two macrocycles [84,86,87].

A mutual influence of the covalently linked porphyrin macrocycles can be controlled, at least formally, by the introduction of different bridging fragments into C_{meso}–C_{meso}, C_{meso}–C_{β}, or C_{β}–C_{β} bonds, for instance: –CH_2– [86–88], –CHOH– [89], –CH_2–CH_2– [86,87,90–93], –CH=CH– [89,91,92,94–102], or –CHOH–CHOH– [96]. Particularly, communication between the porphyrin moieties via linking vinylene (e.g. **4** [94], **5** [96]) or conjugated polyenes –$(CH{=}CH)_n$– [96,103] has been a matter of the special interest.



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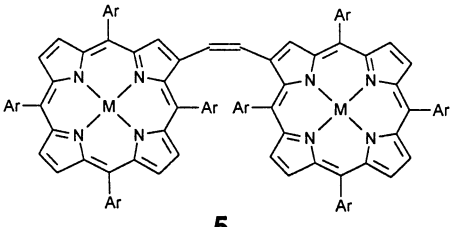

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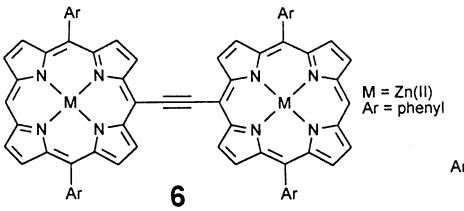
The vinylene bridge can be considered as the simplest example of a covalent connection with its own π-bond(s) that can be conjugated with the linked porphyrin macrocycles, and can facilitate energy and/or electron transfer. The systematic investigations in this direction explored the following bridging units: polyacetylenes [11,41,104–114] (e.g. **6** [11], **7** [11], **8** [108], **9** [110]), aromatic linkers [5,14,15,17,19–27,115–140] (e.g. **10** [14], **11** [123], **12** [128], **13** [115], **14** [115]), a variety of their structural combinations which involve vinylene or acetylene and aromatic moieties [10,28,29,32–36,39,46,50,70,77,106,107,141–184] also in a form of dendrimeric (e.g. **15** [181]) or cyclic architectures.



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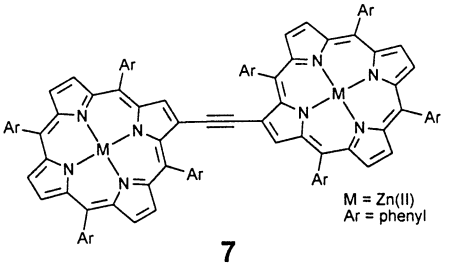

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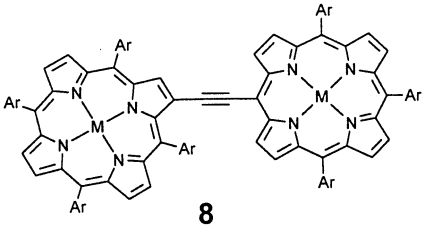

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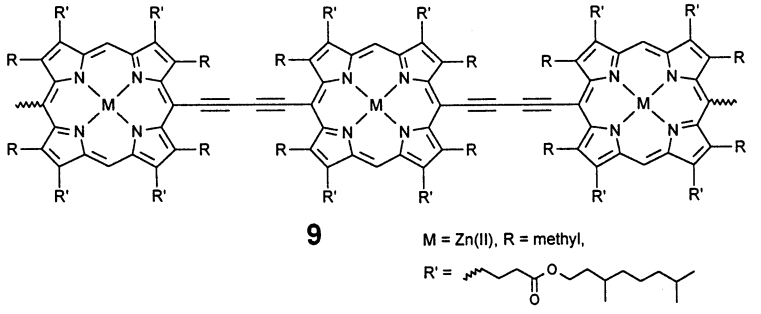

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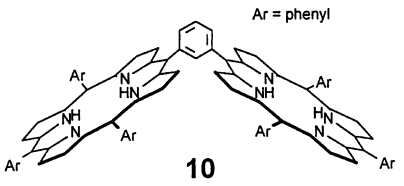

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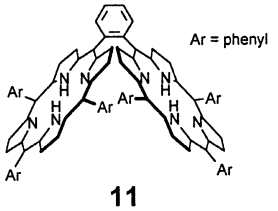

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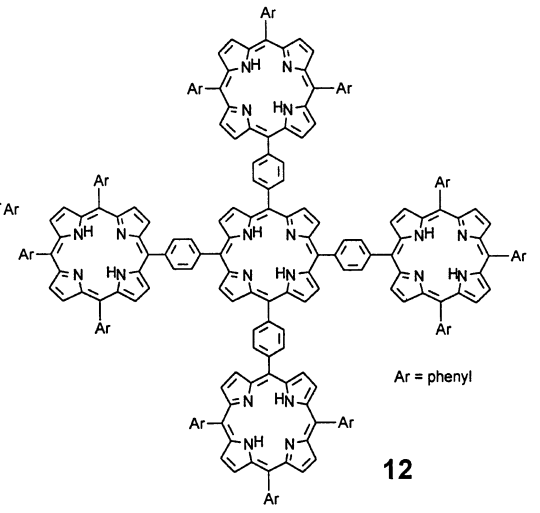

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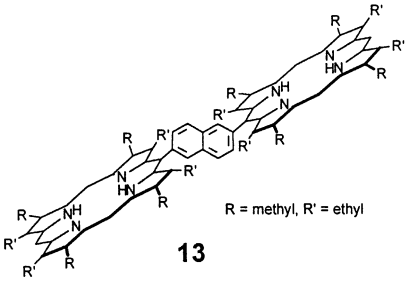

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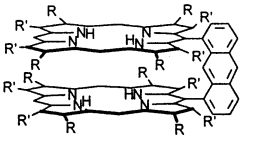

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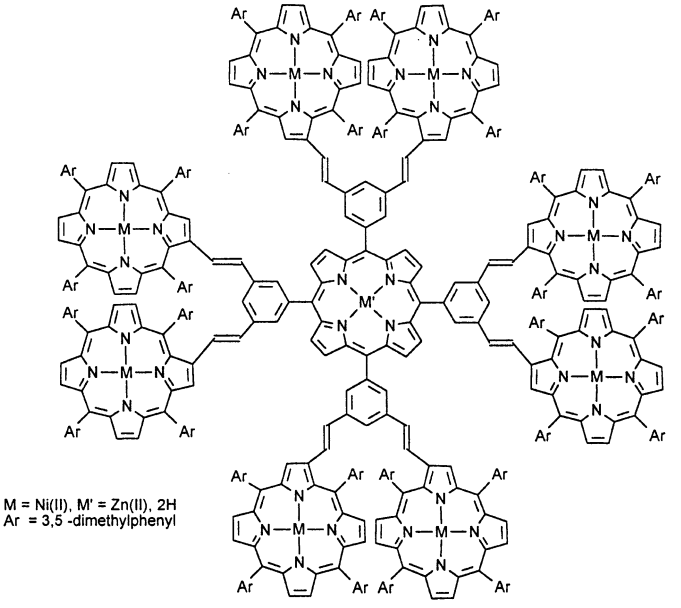

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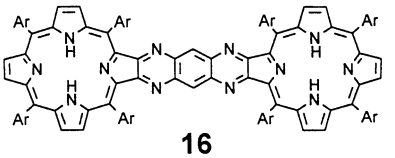
Oligomeric coplanar porphyrin molecules, in which π-systems can interact directly, are of particular interest. Crossley and co-workers described a synthetic approach to rigid, π-conjugated, porphyrin-based molecular wires [30,31,185–192]. These porphyrins **16**, **17**, **18**, **19** are bridged by a planar aromatic fragment of 1,4,5,8-tetrazaantracene. Recently, two other types of conjugated oligoporphyrin systems (**20**, **21**) have been synthesized by Smith and co-workers [193,194].



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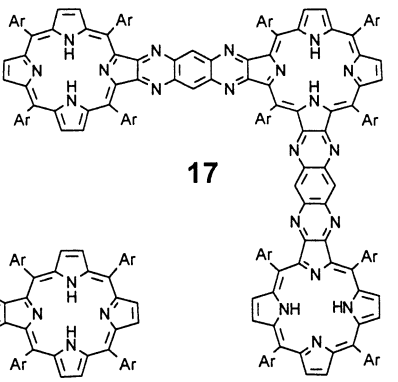

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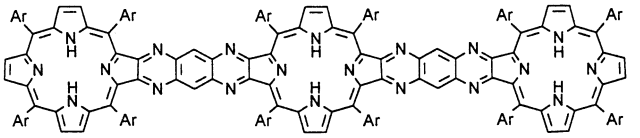

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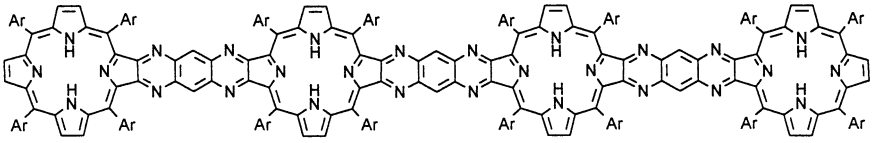

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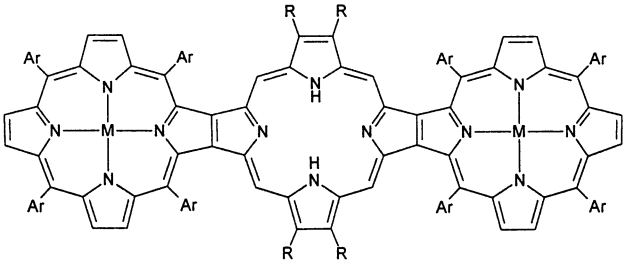
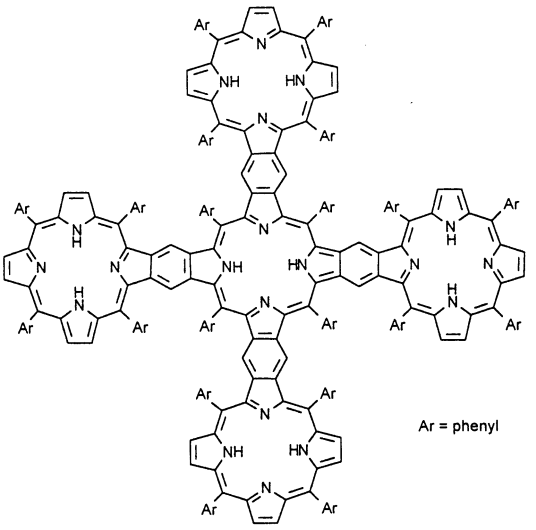
Ar = 3,5-di(*t*-butyl)phenyl



M = Ni(II), Cu(II)
Ar = phenyl, R = ethyl

20



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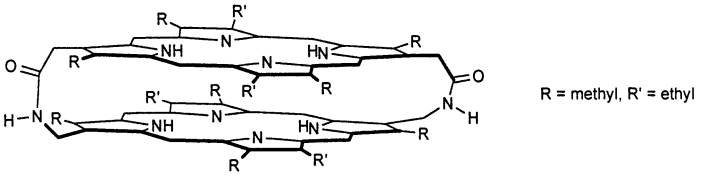
The construction of face to face porphyrins in which two (or more) porphyrin rings are held in parallel conformation (due to rigid or multiple linking, like in **22** [195,196] or **23** [197]), allows the coordinated metal ions to act in concert in catalytic processes such as the reduction of dioxygen or dinitrogen [16–26,38,52,116,124–126,195–207].



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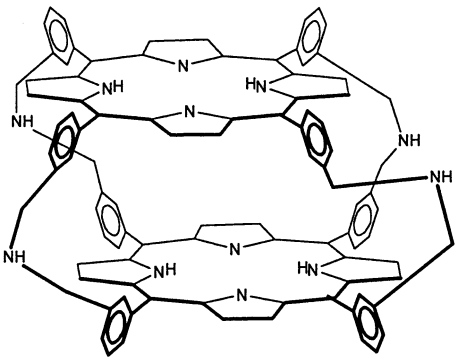

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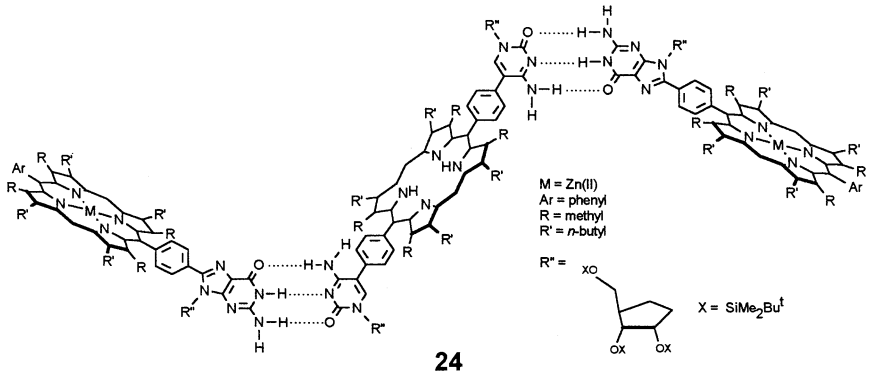
These selected structures (**1**–**21**) present only some representative features for a large variety of oligoporphyrins linked by covalent bridges of different chemical nature, length, and flexibility [3,48,167,208–242].

3. Selected examples of multiporphyrin systems linked by weak interactions

For the sake of comparison, some arbitrarily chosen, but interesting structures that are held together by interactions other than coordination bonds, are described in brief. In particular, hydrogen bonds formed by substituents located on the porphyrin periphery are of special interest [53,217,218,243–253] (e.g. **24** [245], **25** [217], **26** [246], **27** [247]). The electrostatic interactions can also be instrumental in the self-assembly process [254,255]. Finally, π–π stacking between aromatic porphyrin macrocycles results in formation of dimers and oligomers both in solution and in solid state, particularly for derivatives without bulky substituents [110,203,256–262].



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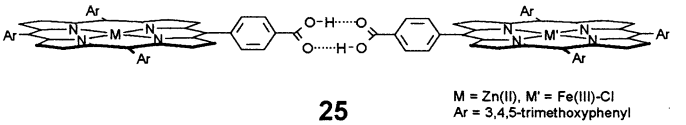

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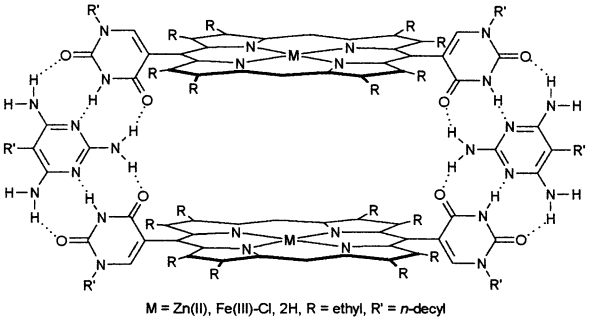

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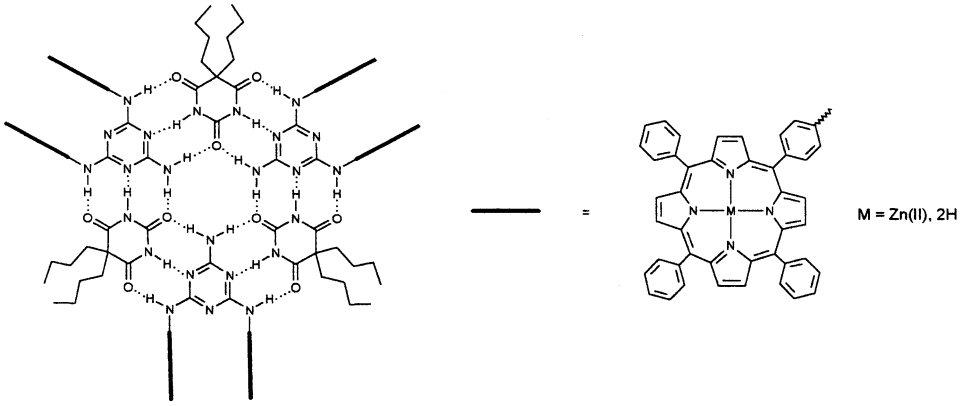

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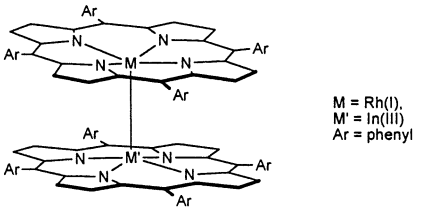
4. Multiporphyrin systems linked by coordinate bonds

4.1. Metal–metal bonds

A peculiar and relatively rare way to create dimeric metalloporphyrins is the formation of a direct bond (of various multiplicity) between the two metal ions at the center of each porphyrin [64,68,124,125,263–268]. Such metal–metal bonded porphyrins may include two like or two different metal centers. Structures of this kind are exemplified by compounds **28** [266] and **29** [124].



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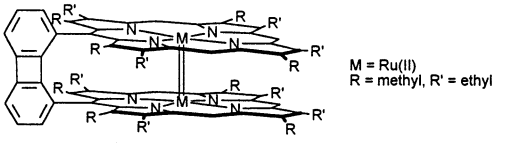

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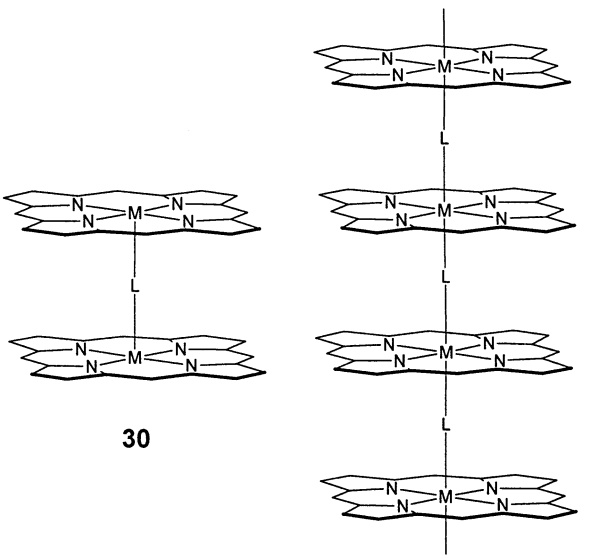
4.2. Multidentate bridging ligands

This separate and very rich class of metallomultiporphyrin architecture uses multidentate ligand as the intermolecular bridge [65–67,69]. Here we present only selected examples of such a construction mode. In most cases, such an approach leads to dimeric structures **30**. The shish-kebab type arrangements [269] provide an example of the polymeric rod type structure **31**.



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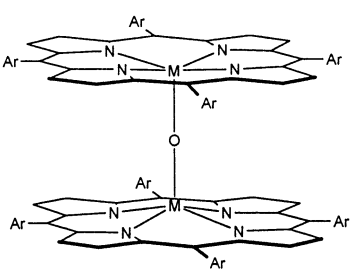
The bridging unit consists frequently of one atom as found in μ-oxo (e.g. **32** [270], **33** [271,272]) [66,270–279], μ-nitrido- [280], μ-carbido- [281,282], and μ-sulfido- [283] dimetalloporphyrin complexes, or in a di-μ-fluorido trimeric gallium complex [284].



M = Fe(III), Ar = phenyl

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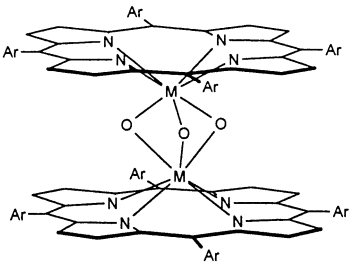
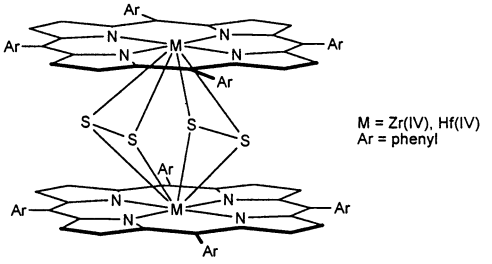


M = Nb(V), Ar = phenyl

33

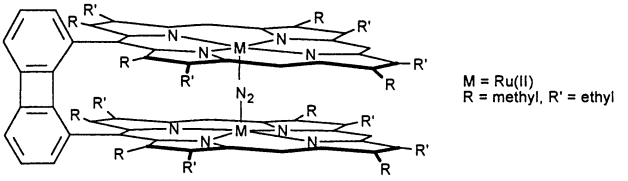
The dimeric and trimeric metalloporphyrins with the μ-hydroxo bridges were also synthesized and structurally characterized [127,285–292]. Very interesting compounds **34** of the general formula $[(TPP)M^{IV}]_2(\mu\text{-}\eta^2\text{-}Q_2)_2$ (M = Zr, Hf, Q = S, Se) contain two bridging ligands S_2^{2-} or Se_2^{2-} [293].



34

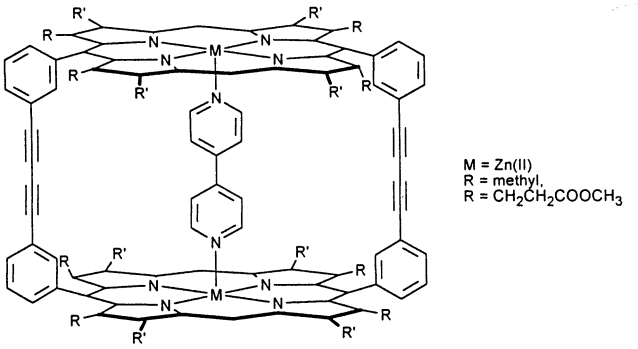
The distance between the porphyrin planes can be controlled by the choice of the bridging ligand as exemplified by the following linking fragments: μ-peroxo- [294,295], μ-cyano- [208], μ-sulfato- [296,297], μ-methanesulfonato- (in polymeric $[(TPP)In^{III}(SO_3CH_3)]_n$) [298], μ-chromato- [299], μ-alkylo- or arylo- [300], μ-(α,ω-diamino)- [215,240,301], μ-(α,ω-diolato)- [42,302], μ-formato- [303], μ-tricyanomethanido- [304], or μ-tetracyanoethenido- [43].

A covalent bond between two metalloporphyrin fragments may impose the appropriate geometry (distance and a mutual orientation) that supports the binding and subsequent coordinate bridging by diatomic molecules: O_2 [16–20], H_2 [19,26], N_2 (compound **35**) [19,24,25].



35

More elaborate bidentate or tridentate ligands have been used as suitable bridges to form polymetalloporphyrin systems. The following bridging molecules have been of interest: imidazolate [126,221,305], pyrazine and its derivatives [40,250,269,306–310], 4,4′-bipyridine (e.g. in **36** [149]) [148–150,153,158,160, 162,167,207,309,311], 2,4,6-tri(4-pyridyl)triazine (e.g. in **37** [148]) [148,149,155, 158,160,162–164,166,170], 1,4-diazabicyclo[2.2.2]octane (DABCO) [163,167,169, 202,204,205,311], 1,4-diisocyanobenzene [67], hydroquinone [312,313], and several other molecules [27,40,44,45,67,122,158,161,166,170,205,215,238,239,249,265,311, 314–318].



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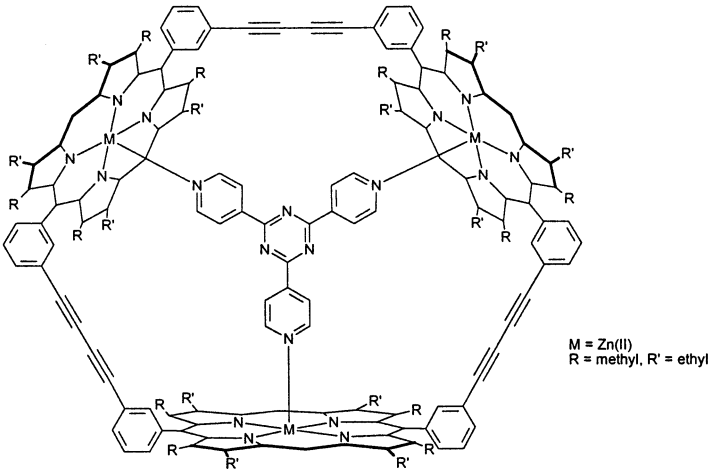
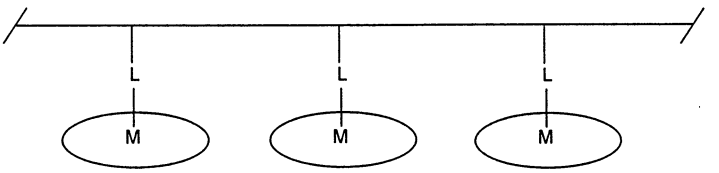


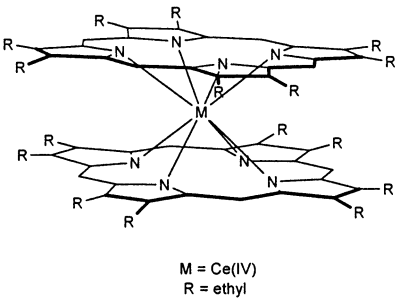
37

Multiporphyrin assemblies **38** in which metalloporphyrins are coordinated by ligands appended to a polymeric backbone [65,69] can be, at least formally, treated as members of this set of complexes.



38

A peculiar class of dimeric and trimeric metalloporphyrins consists of sandwich complexes. In double decker P_2M (e.g. **39** [319]) and triple decker P_3M_2 metalloporphyrins (e.g. **40** [319]), a metal ion serves as a bridging unit between two porphyrins [319–324].



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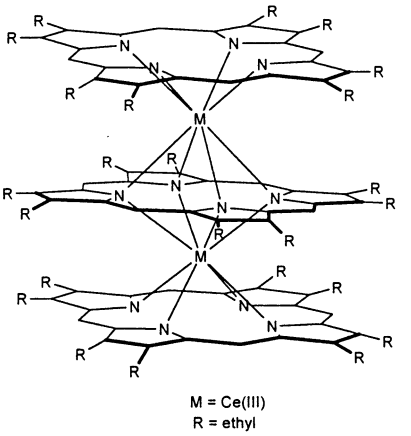


40

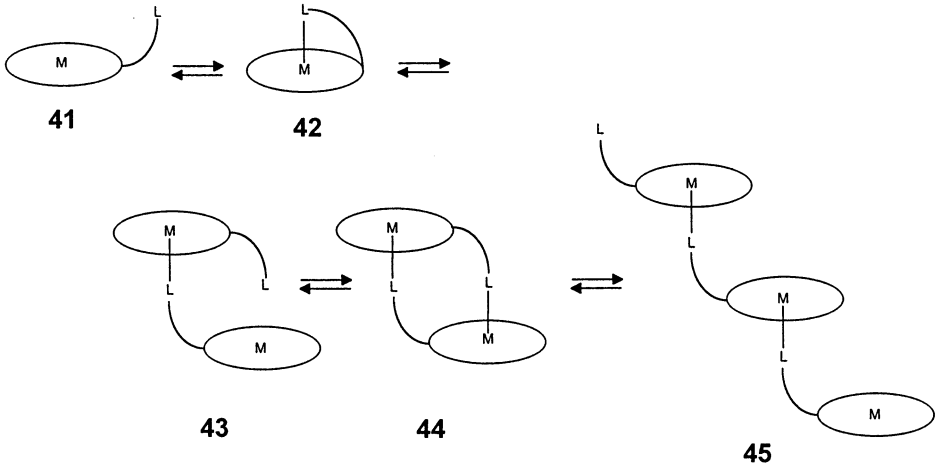
4.3. Self-coordination of hybrid metalloporphyrins

Coordinating properties of porphyrins, widely used in biomimetic chemistry and catalytic studies, can be remarkably modified by introduction of some additional donor groups at the porphyrin periphery. Such substituents are suitable for coordination of the metal ion of the same or other molecular systems. These hybrid bifunctional ligands combine properties of the porphyrin macrocycles with those of the peripheral coordinating moiety.

4.3.1. Chelated hemes

Originally, in biomimetic studies the selection of the appended fragment (imidazole, pyridine, thioalcohol, peptides) was dictated by the coordination environment of the heme in the active center of hemoproteins such as hemoglobin or myoglobin [13,325–329], cytochrome *c* [330], cytochrome *c* oxidase [331], or cytochrome *P*-450 [332,333]. Chelated heme compounds in which the axial ligand is covalently bound to a side chain of heme (**41**) frequently prefer internal coordination (**42**). An appropriate covalent attachment increases the local concentration of the axial ligand and enhances the probability of coordination to the same metal ion without the necessity of a large excess of the external ligand. This approach provides a stoichiometric amount of a built-in nitrogen base. However, in several cases the formation of the dimeric (head-to-tail, open, **43**, or closed, **44**) or polymeric complexes (**45**) accompanied the internal coordination [63,327,328,330,334]. The extent of such dimerization has been correlated with the length and nature of the chelation arm, the coordination properties of the covalently attached base, the

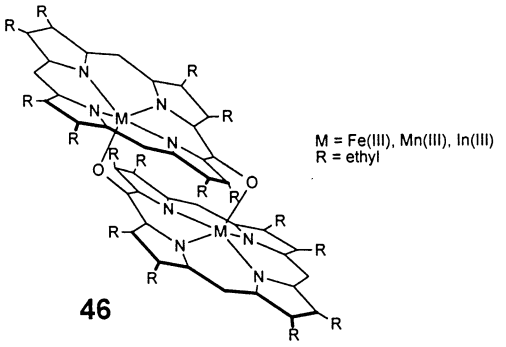
concentration of the chelated heme, the temperature and the presence or absence of an external ligand. For instance, with a short side chain, that covalently appends pyridine to heme, intramolecular binding to form chelated heme was prohibited while external coordination was favored [327]. Traylor et al. demonstrated that such a system exhibits ligand-induced dimerization revealing high degree of cooperativity in the presence of cyanide [327].



4.3.2. Metalloporphyrins with external oxygen donors

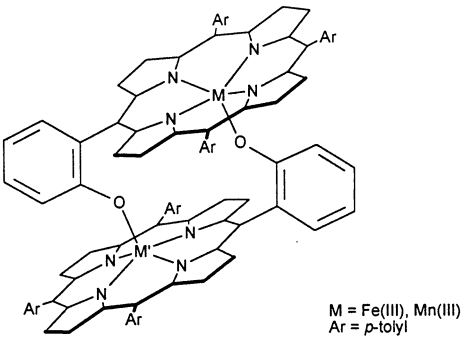
Mono-hydroxy substituted octaethylporphyrin and tetraphenylporphyrin can be considered as representative examples of porphyrin-based polyfunctional ligands. They are able to coordinate through the N_4 core and/or by an ionized hydroxyl group of the periphery. Three structurally distinct locations of the hydroxyl substituent on the basic $OEPH_2$ and $TPPH_2$ pophyrin frameworks are synthetically attainable: at a *meso* position for $OEPH_2$ and at a β-pyrrole or phenyl positions for $TPPH_2$ [335–338].

Iron oxophlorin (*meso*-hydroxyporphyrin) complexes are significant in context of their involvement as intermediates in the conversion of iron porphyrin to biliverdin in the oxidative destruction of heme. Masuoka and Itano demonstrated that insertion of iron into 5-hydroxyoctaethylporphyrin (oxophlorin, $OEPOH_2$) resulted in the formation of the high-spin iron(III) doubly oxo bridged head-to-tail dimer, $[(OEPO)Fe^{III}]_2$, **46** [339]. The properties of $[(OEPO)Fe^{III}]_2$ in solution have been extensively investigated by means of ^{1}H-NMR spectroscopy by Balch and co-workers [340,341]. The ^{1}H-NMR spectroscopic studies indicated that $[(OEPO)Fe^{III}]_2$ undergoes one-electron oxidation without disruption of the dimeric unit to form the monocation $\{[(OEPO)Fe^{III}]_2\}^+$. The ligand-based oxidation occurred and the unpaired electron is delocalized over both macrocycles [342]. The formation of $[(OEPO)Mn^{III}]_2$ was also demonstrated [343]. The structure of the related indium complex $[(OEPO)In^{III}]_2$, which serves as a model for the Fe(III) and Mn(III) analogs, was determined by X-ray crystallography [344].



46

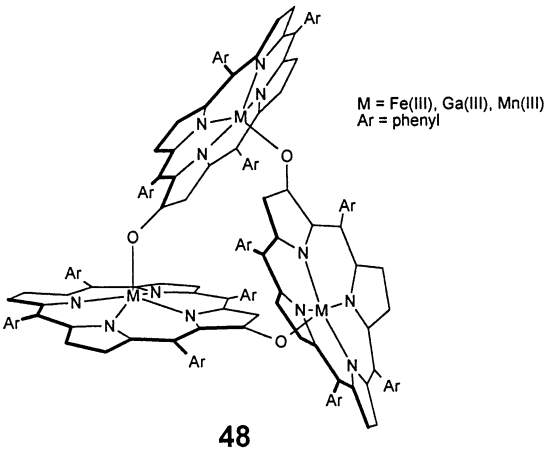
Goff et al. investigated trivalent metal complexes of 5-(2-hydroxyphenyl)-10,15,20-tritolylporphyrin ($TTOPH_2$) [345,346]. The structure of $[(TTOP)Fe^{III}]_2$ (**47**) was determined by X-ray crystallography, and the retention of the dimeric structure in solution was demonstrated by ^{1}H-NMR spectroscopy. The head-to-tail dimerization through phenoxide oxygen was found [345]. Goff and co-workers characterized the manganese(III) dimer $[(TTOP)Mn^{III}]_2$ and detected, by means of ^{1}H-NMR, the diphenoxo bridged heteronuclear complex $\{[(TTOP)Fe^{III}]$-$[(TTOP)Mn^{III}]\}$ [346].



47

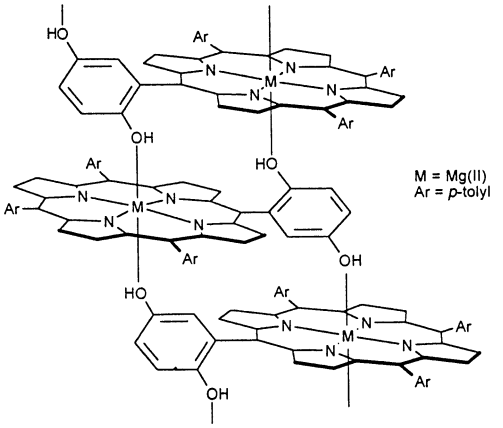
The self-assembly process of a monomeric metal ion(III) 2-hydroxy-5,10,15,20-tetraphenylporphyrin complex $(2\text{-OH-TPP})M^{III}Cl$ (M^{III}: Fe^{III}, Mn^{III}, Ga^{III}) affords the cyclic trimeric complexes **48** of the general formula $[(2\text{-O–TPP})M^{III}]_3$ [347–350]. The ^{1}H-NMR spectroscopic evidence indicated that these compounds have a head-to-tail cyclic trimeric structures with the pyrrolic–alkoxide groups forming bridges from one macrocycle to the metal ion in the adjacent macrocycle. The inherent optical asymmetry of the basic $(2\text{-OH–TPP})M^{III}$- unit leads to formation of the racemic mixture of enantiomeric metal ion(III) porphyrin trimers. The structure of $[(2\text{-O–TTP})Fe^{III}]_3$ has been determined by X-ray crystallography [350]. The porphyrinic skeleton of $[(2\text{-O–TTP})Fe^{III}]_3$ constitutes a common pattern for

these homometallic trimers. A similar cyclic structure was found also for heterometallic trimeric iron(III), gallium(III) and manganese(III) complexes of 2-hydroxytetraarylporphyrin that were detected by ^{1}H-NMR and mass spectrometry investigations [350].



48

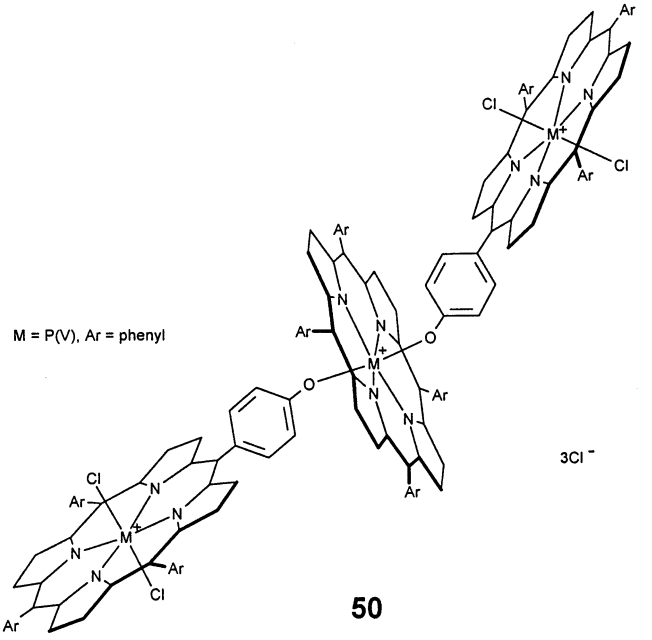
^{1}H-NMR studies of magnesium(II) 5-(*p*-hydroquinonyl)-10,15,20-tritolylporphyrin complex performed by Miyaji et al. demonstrated self-assembling through two oxygen-magnesium bonds (structure **49**) [351]. The extent of polymerization depends on the temperature and concentration, although the exact value of an aggregation number was not determined.



49

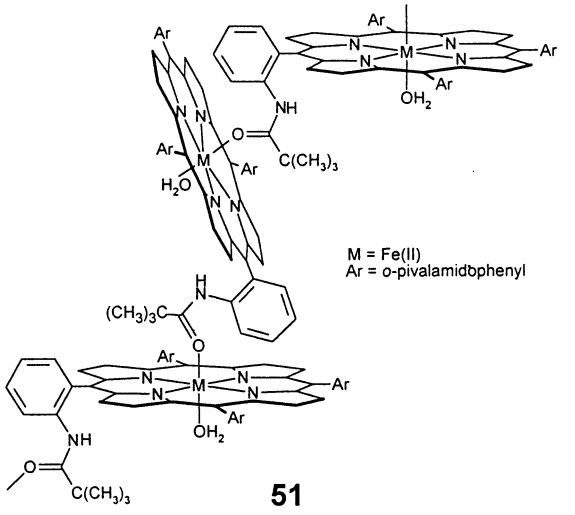
A similar external binding motif of a hybrid porphyrin, i.e. a phenoxy group located at the *meso*-substituent of porphyrin was used to construct center-to-edge porphyrin dimers and trimers (**50**), although the porphyrin center contained a metalloid instead of a metal ion [352,353]. A covalent connection between the

central phosphorus atom which was surrounded by one porphyrin, and the phenoxy groups at the *meso* position of two other porphyrins held the system together.



50

The crystal structure of *meso*-α,α,α,α-tetrakis(*o*-pivalamidophenyl)porphyrinato iron(II) contains polymeric chains of metalloporphyrins (**51**) with the Fe atom of one unit coordinated to the pivalamide oxygen of the next unit [354].



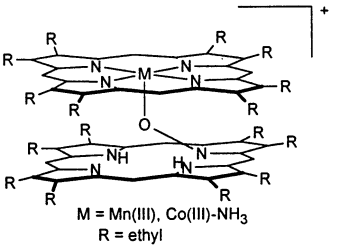
51

Senge and Smith determined a head-to-tail dimeric structure of bis(anhydro-*meso*-rhodochlorinato-XV methyl ester)zinc(II) [355]. The presence of fused cyclohexanone rings resulted in the formation of the dimer, consisting of two

macrocycles related to each other by an inversion center and held together by a coordination bond from a carbonyl oxygen of one macrocycle to the zinc(II) ion of another macrocycle.

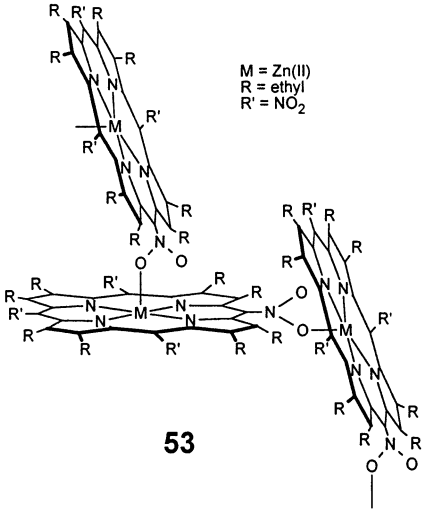
The crystal structure was reported for a zinc(II) complex of 2,8,12,18-tetraethyl-3,7,13,17 - tetramethyl - 5,15 - {1,5 - naphtylenebis[[[[[[(oxyethylene)oxy]ethylene]oxy]-ethylene]oxy]phenyl]}porphyrin, a porphyrin strapped with a dibenzo-crown ether. Oligomers, in which the zinc ion of one macrocycle is bound to O(7) of the crown ether strapped over a second unit, were found in this structure [356].

Dimeric cationic complexes **52** were obtained in which the N-oxide of octaethylporphyrin served as a monodendate, O-bound axial ligand to manganese(III) or cobalt(III) ions with a normal octaethylporphyrin molecule [357]. The corresponding X-ray structures demonstrated that the two macrocycles are nearly parallel in these assemblies, with a relatively short separation between the porphyrin planes (3.34 Å).



52

Zinc(II) 2,3,7,8,12,13,17,18-octaethyl-5,10,15,20-tetranitroporphyrin contains a markedly distorted macrocycle due to sterically hindered peripheral substituents. It also presents a noteworthy crystal structure that demonstrates the ligation of *meso* nitro groups to the zinc(II) center (**53**). The two neighboring molecules are almost orthogonal to each other [358].

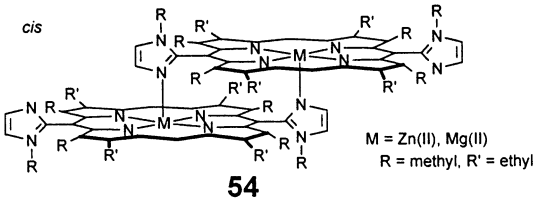


53

Several model systems have been designed to mimic the aggregation behavior of bacteriochlorophylls and chlorophylls that is observed in vivo [359]. Structures, utilizing a coordination bond were considered in these investigations. For example, methyl bacteriochlorophyllide *d* forms a stable dimer in a chloroform solution [360,361]. The head-to-tail models with the hydroxyl group of the 1-hydroxyethyl substituent coordinated to the magnesium(II) ion of the adjacent molecule were in agreement with the ^{1}H-NMR data.

4.3.3. Imidazole, pyrazole and amine appended metalloporphyrins

In an approach to construct rigid dimers based on a self-organization via coordination bonds several ligands were tested. For instance, introduction of *N*-methylimidazole into the 5,15-positions of 2,8,12,18-tetraethyl-3,7,13,17-tetramethylporphyrin resulted in a hybrid porphyrin ligand with two additional centers of coordination [362,363]. The porphyrin and its zinc(II) and magnesium(II) complexes demonstrated the existence of two atropisomers which were differentiated by the mutual positions of two *N*-methyl substituents (*cis*-located on the same side of the porphyrin plane, *trans*-on two opposite sides). The zinc(II) complex of this porphyrin formed a slipped-cofacial dimer revealing a head-to-tail structure (**54**, **55**) [362,363]. An analogous arrangement was found for the corresponding magnesium(II) complex, although in this case a tendency for dimerization was definitely higher for the *cis*-atropisomers. The *cis*-configuration prohibited a further polymerization. In contrast, the trimeric complex **56** was also observed when the magnesium(II) *trans*-porphyrin was used as a building block. The trimerization, found for magnesium(II), but not for zinc(II) complexes, was explained by the higher preference of magnesium(II) for six-coordination [363].



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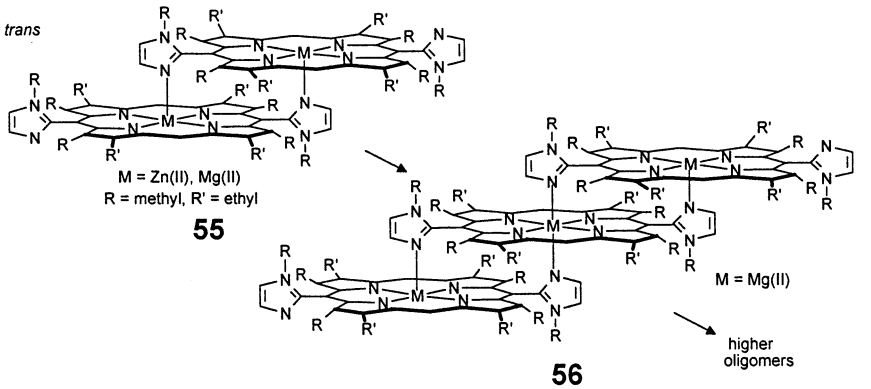
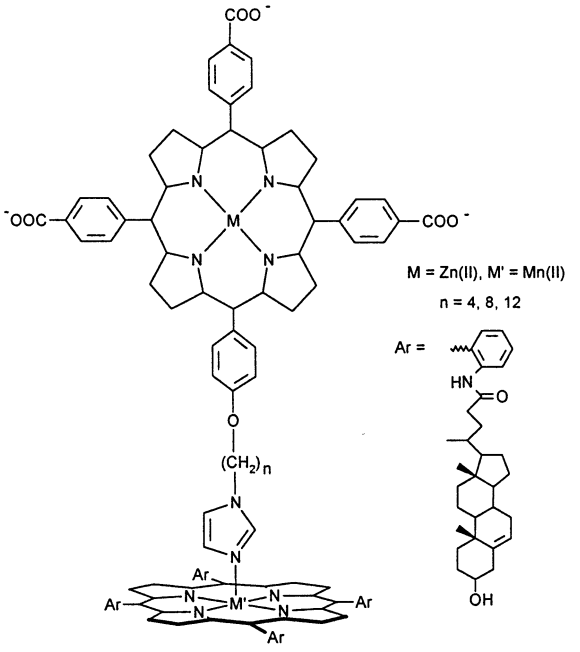


55 **56**

A spectacular application of the multi-porphyrin self-assembly was presented by Groves and co-workers in their effort to design and construct a model system to probe the nature of the electron transfer between cytochrome *c* and a redox center embedded within a phospholipid bilayer. A trianionic zinc porphyrin binds to cytochrome *c* at the membrane surface, while being coordinated to a membrane spanning manganese(II) porphyrin (only this diporphyrinic fragment **57** is shown) via an appended imidazole [364,365].



57

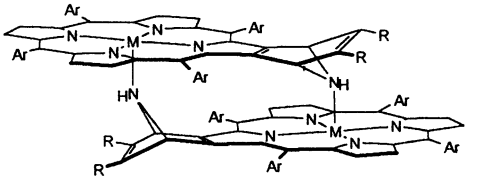
Bied–Charreton and co-workers synthesized derivatives of tetraphenylporphyrin bearing *N*-imidazolyl or a primary amino group attached by alkyl chain varying from three to five methylene groups to the *ortho* or *meta* position of one of the *meso*-phenyl groups [366,367]. The zinc(II) and cobalt(II) complexes of an amino appended porphyrin and the cobalt(II) complexes of an imidazole appended porphyrin, both with the appendages at a *meta* position of one of the *meso*-phenyl groups, demonstrated the formation of the cyclic head-to-tail dimers [366,367]. The localization of the appended chain in the *ortho* position resulted in the monomer–dimer equilibrium. The monomeric complex is characterized by the internal coordination of the covalently bound nitrogen base [366], which corresponds to the structure **42** of the previously discussed chelated hemes.

Recently, the analysis of ^{1}H-NMR data demonstrated that, in a chloroform solution, the monomeric zinc(II) complex of 5-(3-pyrazolyl)-10,15,20-tritolylporphyrin exists in equilibrium with a cyclic head-to-tail trimer held together by

coordination of the pyrazolyl nitrogen atom from one macrocycle to a zinc(II) ion of the adjacent macrocycle [368].

Hexa- and dodecaporphyrin systems were formed by anchoring iron(III) protoporphyrins by histidine residues of polypeptide chains covalently attached to another porphyrin [369,370].

Zinc(II) porphyrin with a fused 7-azabicyclo[2.2.1]heptadiene ring was shown to form a cyclic dimer **58** in solution [371]. This complex crystallizes as a cyclic hexamer with Zn(II) ions coordinated to the secondary amino nitrogen atom of a neighboring unit.



58

In a recent work, Hunter and coworkers investigated a self-assembly process of zinc(II) *meso*-aminophenylporphyrins [372]. Cyclic dimers were formed by *ortho*- and *meta*-NH_2 substituted systems, while the *para*-substitution led to an open chain oligomer.

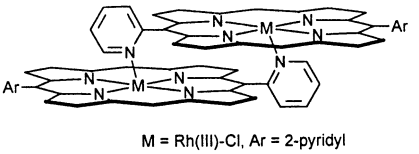
4.3.4. Pyridine appended metalloporphyrins

Several polymeric or oligomeric structures based on a self-assembly approach have been constructed using metalloporphyrin–pyridine interaction[1]. Pyridine acts as a versatile ligand toward a variety of metal ions, and its well-known coordinating properties can be easily adopted to construct self-assembled systems. From the synthetic point of view, it is very important that pyridine can be easily functionalized. Consequently, it can be readily built into the porphyrin *meso*-position(s). The use of mixed *meso*-substituted pyridyl/phenyl porphyrins as linkers for metal ion centers can provide the connection to as many as four metal centers by coordination of the *meso*-pyridyl groups. In addition, the pyridine ring offers three available carbon positions through which it can be attached to a porphyrin. This variability provides an additional route to control the spatial architecture of designed assemblies.

Insertion of zinc(II), rhodium(III), magnesium(II), or ruthenium(II) into 5-(2-pyridyl)-10,15,20-trisubstituted porphyrins resulted in cyclic dimeric complexes (e.g. **59** [373]) [373–376]. For Zn(II) complexes, it was shown that such edge-over-edge structure is retained after a reduction to a zinc(II) bacteriochlorin dimer [377]. Two

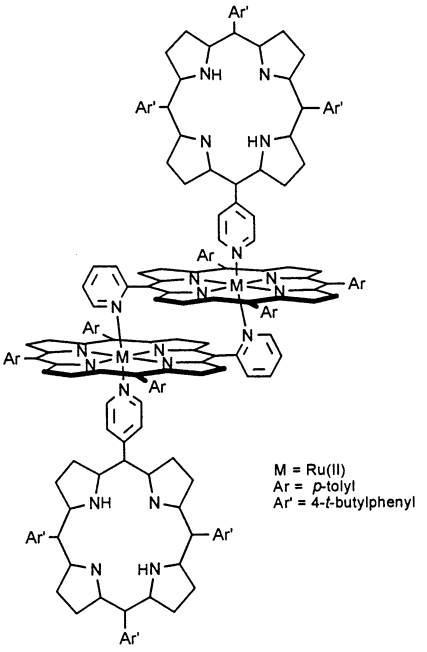
[1] Several aspects of metallopyridylporphyrin self-assembly have been also covered in the review article by T. Imamura and K. Fukushima, Coord. Chem. Rev. 198 (2000) 133.

additional mono-*meso*-(3-pyridyl) or (4-pyridyl) substituted porphyrin ligands can coordinate to Ru(II) ions of the cyclic dimer to form tetrameric assemblies **60** [377].



M = Rh(III)-Cl, Ar = 2-pyridyl

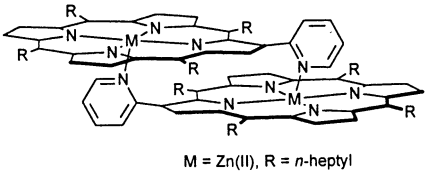
59



M = Ru(II)
Ar = *p*-tolyl
Ar' = 4-*t*-butylphenyl

60

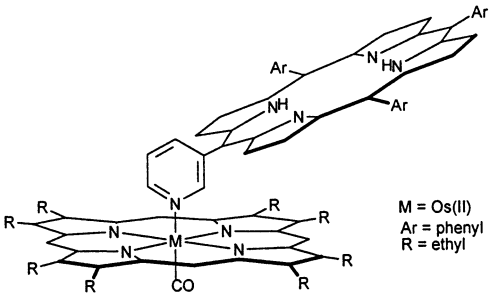
The zinc-coordinating pyridine function can be also located at a β-position as in 2-(2-pyridyl)-5,10,15,20-tetra-*n*-heptylporphyrin [374]. The stacked structure with the pyrrole-*meso* overlap has been suggested for the resulting dimer **61**. The zinc(II) complex exists predominantly in a dimeric form in chloroform-*d* solution but remains in dynamic exchange with a monomeric species [374].



M = Zn(II), R = *n*-heptyl

61

Coordination of 5-(3-pyridyl)-10,15,20-triphenylporphyrin or 5,10-bis(3-pyridyl)-15,20-diphenylporphyrin to osmium(II) complex of octaethylporphyrin leads to oblique-type dimeric (**62**) and trimeric (**63**) complexes, respectively [378]. ^{1}H-NMR data show that the latter exists as a mixture of two atropisomers. Recently, Alessio et al. reported the synthesis of an analogous ruthenium(II) dimer formed in reaction of (TPP)Ru^{II}(CO) with mono-(3-pyridyl)-substituted porphyrin [379,380], and a pentamer from four (TPP)Ru^{II}(CO) molecules coordinated to the central zinc(II) 5,10,15,20-tetrakis(3-pyridyl)porphyrin [380].



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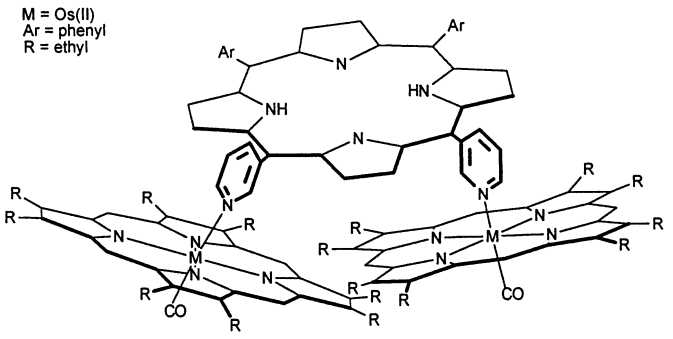
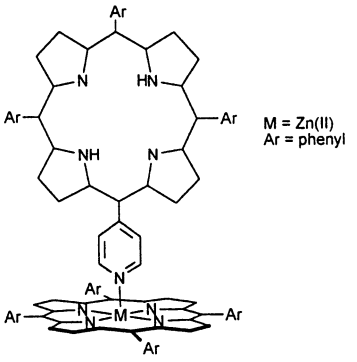
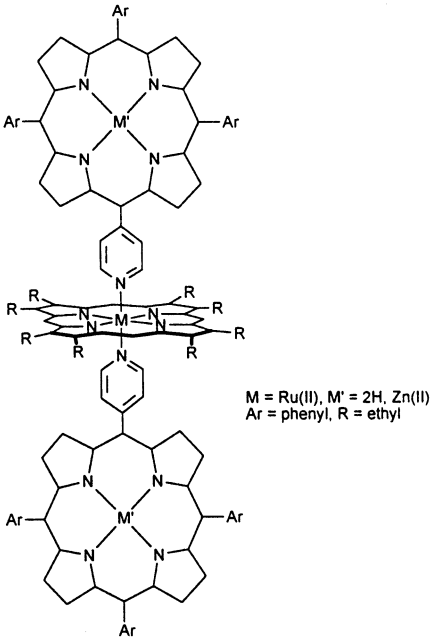


63

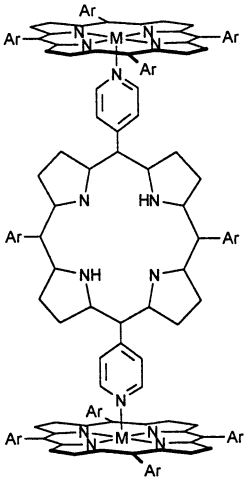
Numerous polymeric or oligomeric complexes with 4-pyridyl/phenyl porphyrins have been designed and characterized. Such a localization of pyridine nitrogen atom results in a perpendicular arrangement of neighboring macrocycles. Here the possible structures are exemplified by representative oligomeric complexes of zinc(II) [381–384], ruthenium(II), and osmium(II) [49,385–390], **64** [382], **65** [385,387], **66** [382], **67** [388], **68** [388], **69** [390], **70** [389], and a zinc(II) porphyrin zigzag polymer **71** [381,382].


M = Zn(II)
Ar = phenyl

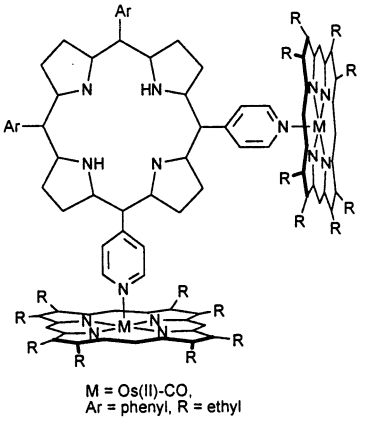
64


M = Ru(II), M' = 2H, Zn(II)
Ar = phenyl, R = ethyl

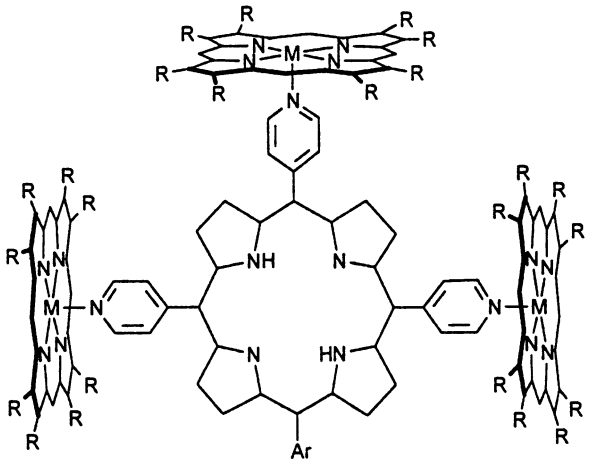
65


M = Zn(II)
Ar = phenyl

66

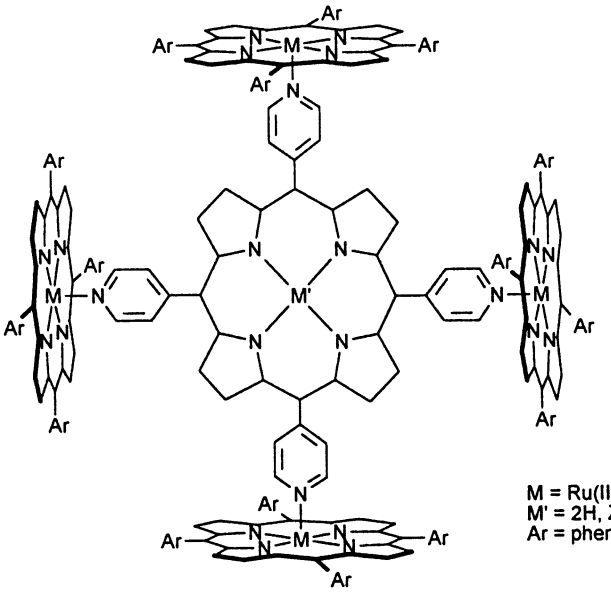

M = Os(II)-CO,
Ar = phenyl, R = ethyl

67



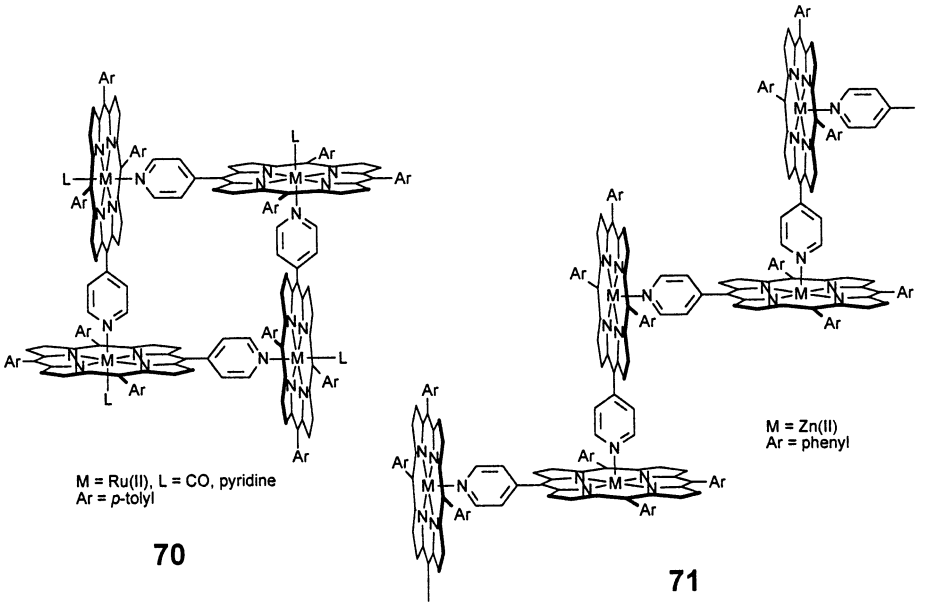
M = Os(II)-CO,
Ar = phenyl, R = ethyl

68



M = Ru(II)-CO
M' = 2H, Zn(II)
Ar = phenyl

69



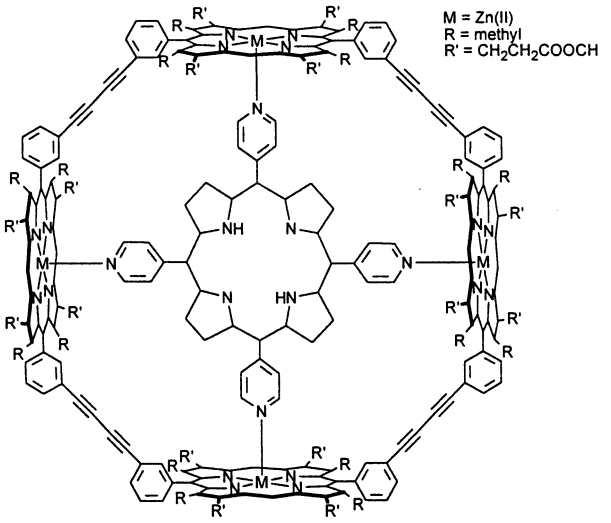
70 **71**

Aggregation patterns for zinc(II) tetra(4-pyridyl)porphyrin in the solid state were determined using X-ray crystallography [391,392]. These complexes form two types of coordination polymers through ligation of the peripheral pyridyl of one molecule to the zinc(II) ion of the adjacent zinc(II) porphyrin. The first type contains one-dimensional chains with a zigzag conformation (similar to **71**); in the second group three-dimensional complex polymeric structures are found. The mutual arrangement of the zinc(II) porphyrin building blocks was related to the choice of solvents used for crystallization [391,392].

A hybrid, two-dimensional polymeric network is formed by coordination of 5,10,15,20-tetrakis(4-pyridyl)porphyrin to a manganese(III) tetraphenylporphyrin [384].

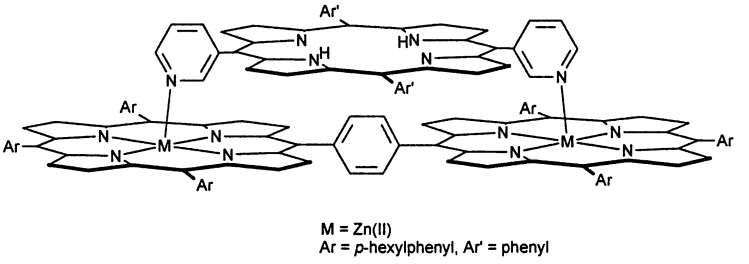
5,15-Bis(4-pyridyl)porphyrin and 5,10,15,20-tetrakis(4-pyridyl)porphyrin were shown to bind within the cavity of the cyclic zinc(II) oligoporphyrins [49,150,153,157–160,162,205,393]. These structures are exemplified by the representative complex **72** [150,157] composed from the host tetramer and 5,10,15,20-tetrakis(4-pyridyl)porphyrin. 4-Pyridyl/phenyl porphyrins also served as efficient catalysts for the cyclization process [150,153,159,160].

5,15-Bis(4-pyridyl)porphyrin and 5,10,15,20-tetrakis(4-pyridyl)porphyrin were found to form 1:1 and 1:2 complexes, respectively, with a rigid dizinc(II) bis-porphyrin with U-type geometry [394].



72

The combination of zinc(II) diporphyrin linked by a *p*-phenylene group, and free-base porphyrins bearing two or four *meso* 3-pyridyl or 4-pyridyl substituents produced assemblies containing three or five porphyrinic fragments, e.g. **73**–**76** [131–137].



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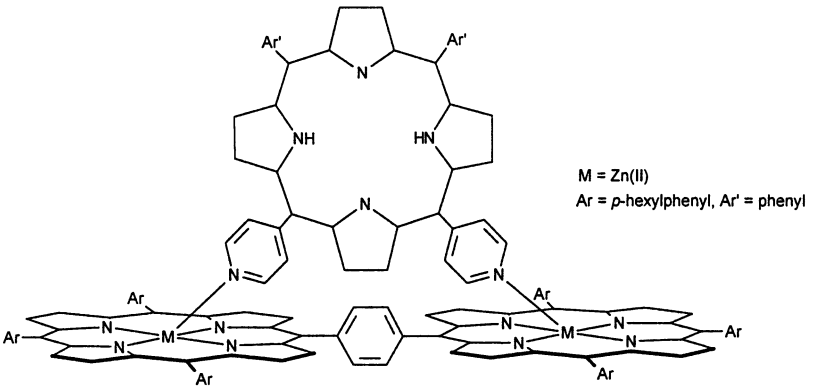

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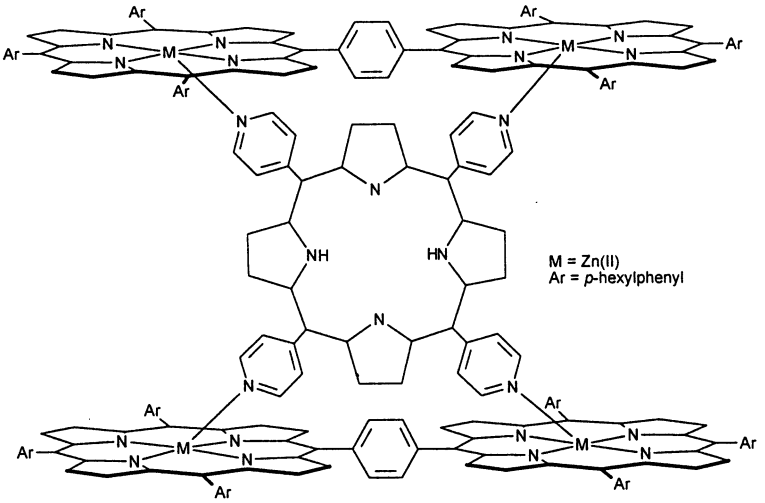

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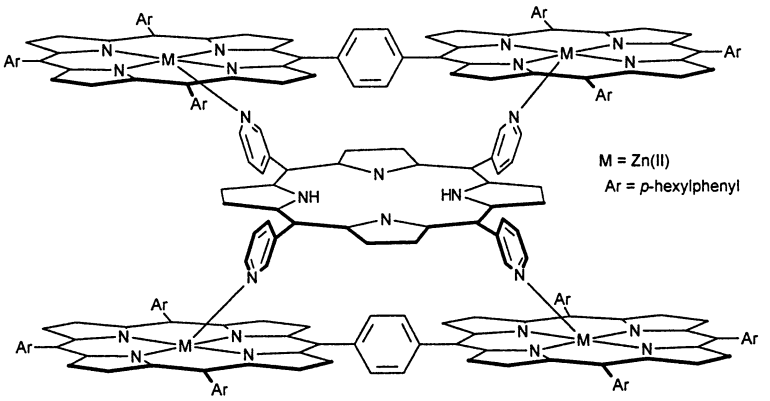


76

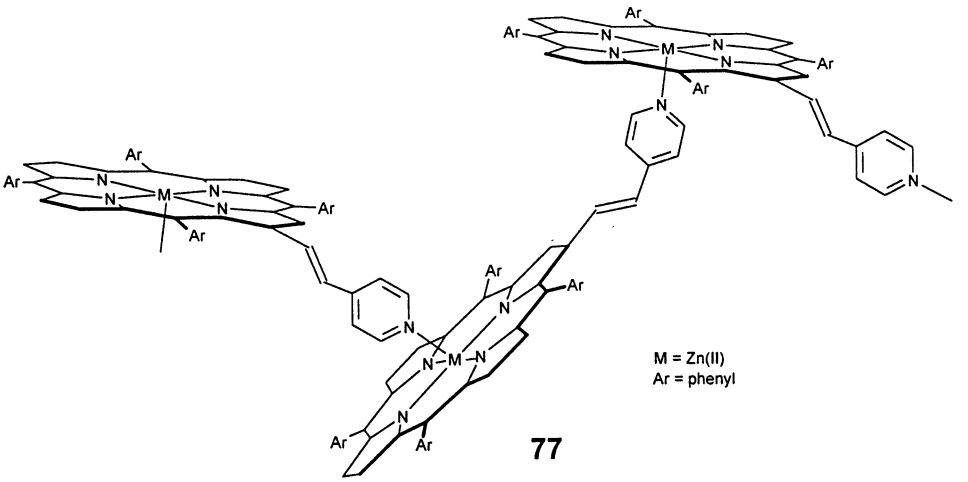
Recently an approach to porphyrin dendrimers was demonstrated that was based on a coordination of *meso*-(4-pyridyl) substituents of porphyrinic dendrons to a ruthenium(II) porphyrin [171]

Coordination of *meso*-(4-pyridyl)porphyrinato tin(IV) to a zinc(II) porphyrin can be supported by binding of the Sn(IV) ion to carboxylic functions, which are covalently attached to the Zn macrocycle. A similar simultaneous coordination was used to construct a trimer of two zinc(II) porphyrins and tin(IV) bis(4-pyridyl)porphyrin [395].

Pyridyl substituents can be also attached to the porphyrin macrocycle with the use of appropriately designed spacers.

Cis/*trans* isomerization of a vinylene bridge linking 4-pyridyl group to a β

position of zinc(II) tetraphenylporphyrin changes the organization of porphyrinic units: a coordination polymer (**77**) with *trans* configuration can be converted to a cyclic dimer (**78**) by UV irradiation [396].



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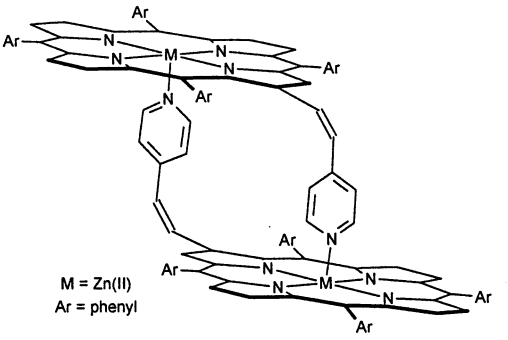
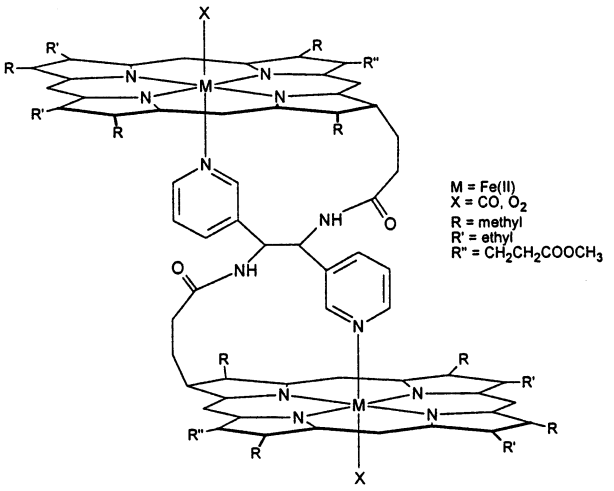


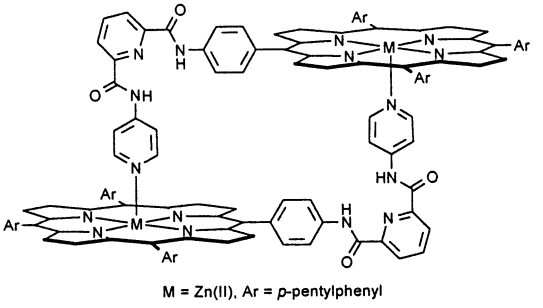
78

In their search for spectroscopic and functional models of hemoglobin, particularly for cooperativity investigations, Traylor and co-workers synthesized a novel example of a dimeric structure **79** (a symmetric diheme) where two iron porphyrins are simultaneously linked through a covalent bridge that contains two pyridine fragments which are also coordinated to the iron(III) centers [13,326].



79

Hunter and co-workers designed a series of zinc(II) porphyrins with a covalently appended pyridine binding center. They investigated the self-assembly processes leading to cyclic structures using the zinc(II)–pyridine coordination as the major linkage [51,223,397,398]. The design of the appended arm geometry lead to dimeric (**80**), trimeric (**81**), or tetrameric architecture (**82**), respectively [51,397,398]. A two-component system **83** containing three porphyrins was also prepared [223].



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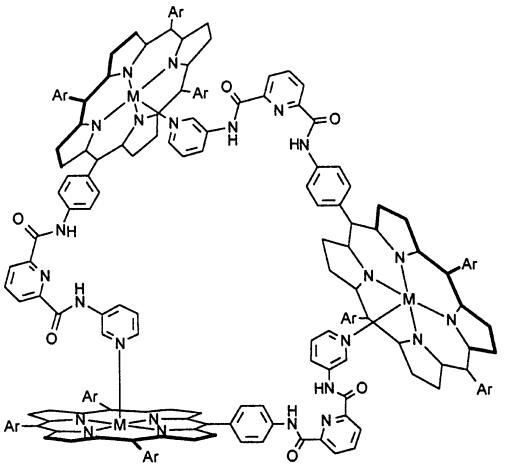
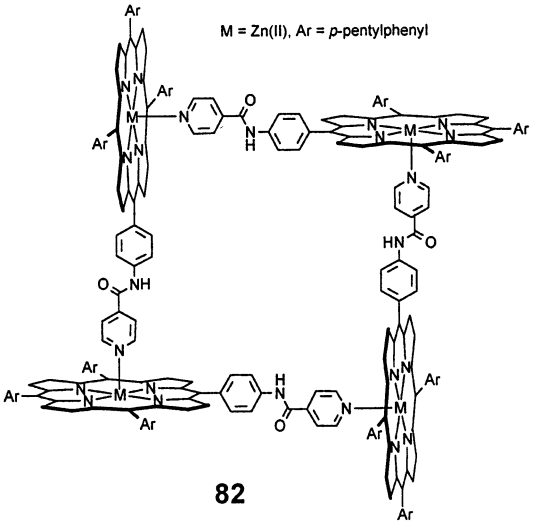


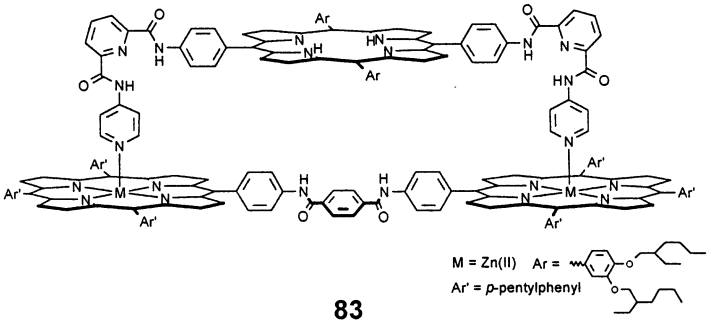
M = Zn(II), Ar = *p*-pentylphenyl

81



M = Zn(II), Ar = *p*-pentylphenyl

82

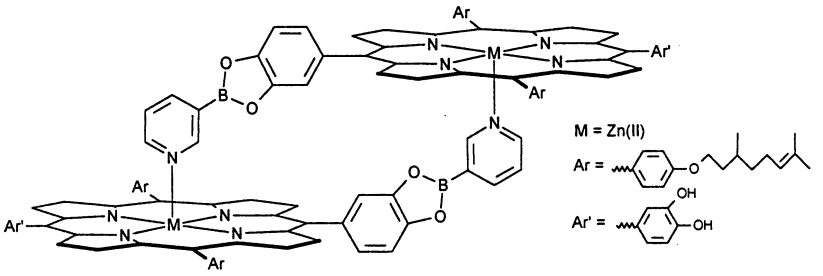


M = Zn(II) Ar =

Ar' = *p*-pentylphenyl

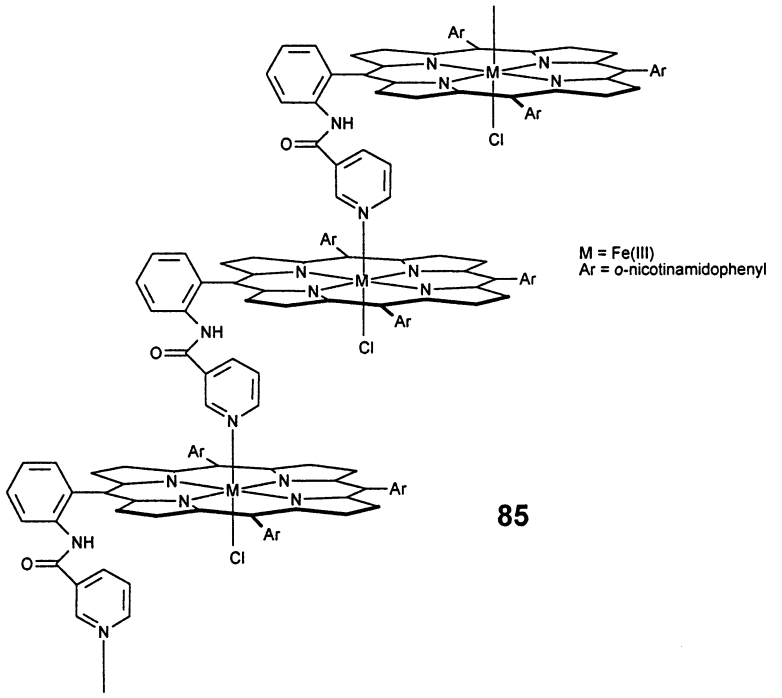
83

Shinkai and co-workers demonstrated the generation of new porphyrin-containing supramolecular assemblies by the combination of two reversible processes, i.e. pyridine–metal ion coordinate bond formation and boronic acid–diol interaction [399]. Thus a 1:1 mixture of zinc(II) dicatechol porphyrin and pyridine-3-boronic acid self-assembles into dimer **84**. Analogous synthesis using magnesium(II) dicatechol porphyrin resulted in a polymeric structure [399].



84

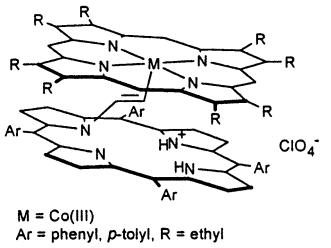
It is worthwhile noting that the iron(III) complex of the potentially binucleating ligand, *meso*-α,α,α,α-tetrakis(*o*-nicotinamidophenyl)porphyrin demonstrated one of the very first examples of the polymeric structure **85** with the pyridine–iron(III) bond as the linking motif. The polymeric structure of the complex consists of infinitive chains of metalloporphyrins with the pyridine of a nicotinamide picket of one unit coordinated to the iron(III) center of another [400].



85

4.3.5. Other types of appended coordination centers

Setsune et al. prepared a series of vinylene-Co, *N′*-bridged bis- (e.g. **86**), tris- and tetrakisporphyrins (e.g. **87**), in which an organometallic linkage was used to connect free-base porphyrins and cobalt(III) complexes [401].



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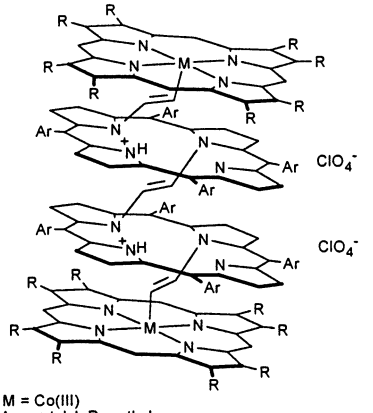


87

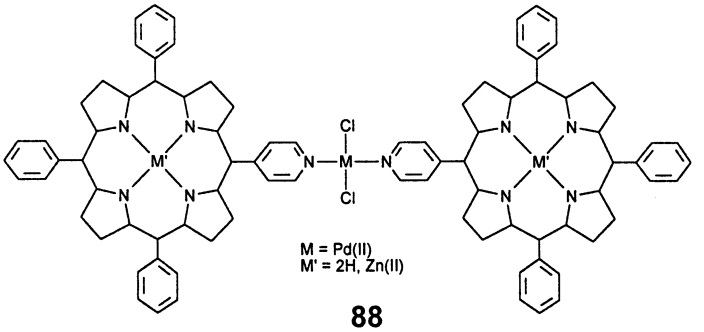
Under suitable conditions, zinc(II) 5,10,15,20-tetrakis(4-cyanophenyl)porphyrin forms two-dimensional coordination polymers through direct ligation of two cyano groups of each complex to the metal center of two neighboring molecules [402].

X-ray crystallography shows that the zinc(II) complex of a cyclopropanyl-annulated chlorin utilizes the attached isocyanide function to give a centrosymmetric dimer linked by two coordination bonds [403].

4.4. Linkage by external metal centers

The covalently appended ligand(s) may coordinate internally (to form a chelated heme) or, as it was previously demonstrated, they can play an essential role in polymerization (oligomerization) process while coordinating to the metal ion of another subunits. Here selected examples are presented where the coordination involves the external metal ions not built into the porphyrin macrocycles and, consequently, the external metal ion is in position to bring together two or more metalloporphyrin subunits. Frequently the arrays are generated by a self-assembly approach. In principle, such a methodology provides access to one-, two- and three-dimensional architectures that are based on porphyrin and metalloporphyrin subunits.

Dimers (e.g. **88**–**90** [404]) and tetramers (**91**, **92** [404], **93** [405]), including the porphyrinic molecular squares, are formed by self-assembly process which involves the metal ion coordination (Pt^{II}, Pd^{II}, Ru^{II}) to the peripheral pyridyl group of the mixed *meso*-(4-pyridyl)/phenyl porphyrins [404–409]. Recently this approach was used with success to bring together different types of porphyrin subunits to give linear tapes (**94**, **95**), and a spectacular square array of nine macrocycles (**96**) [410]. Mono(4-pyridyl)porphyrins and Pd(II) organometallic complexes were also applied to construct a group of dendrimers (for example, **97** and **98**) containing up to 12 porphyrinic macrocycles [411].



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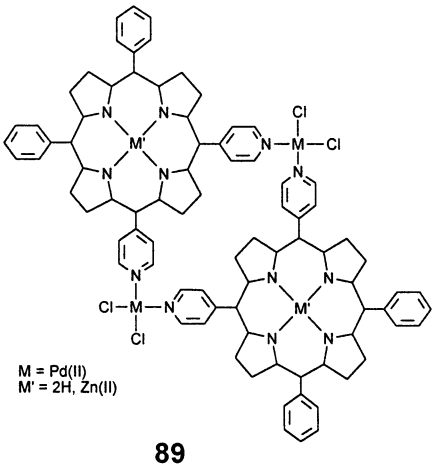

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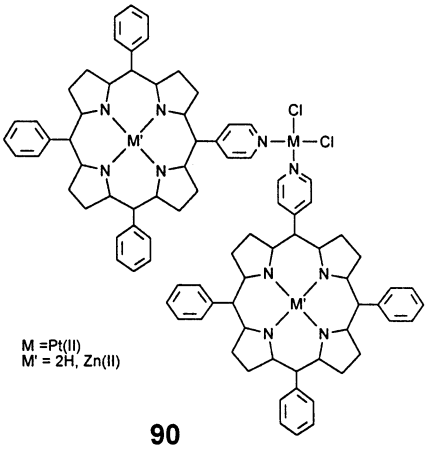

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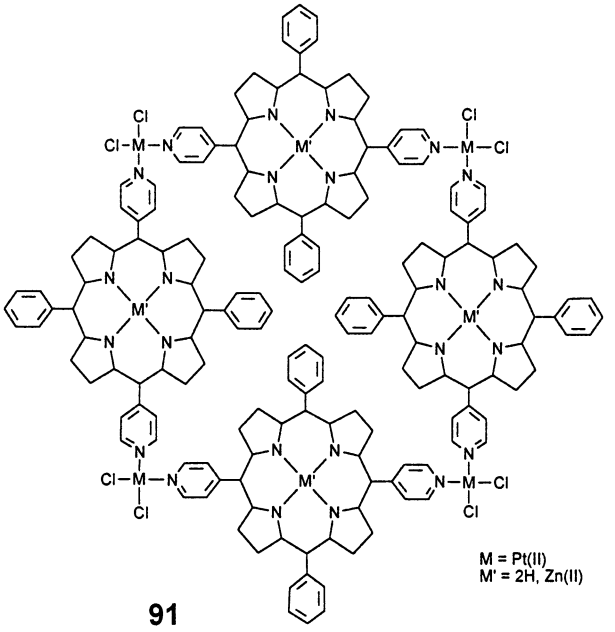

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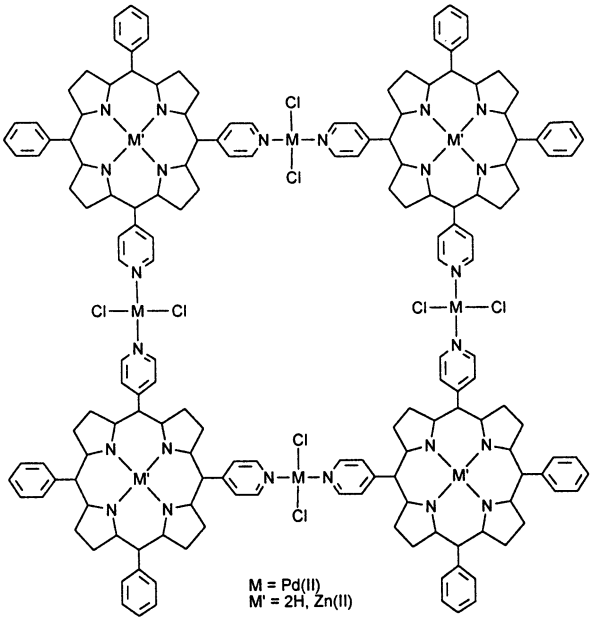

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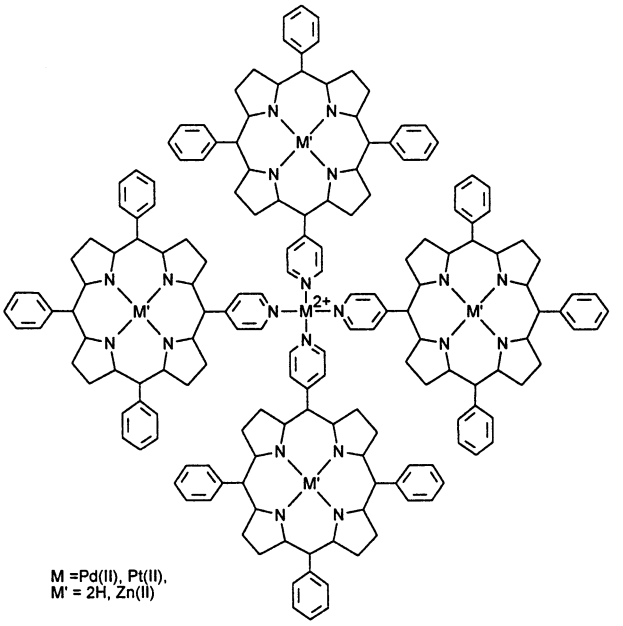

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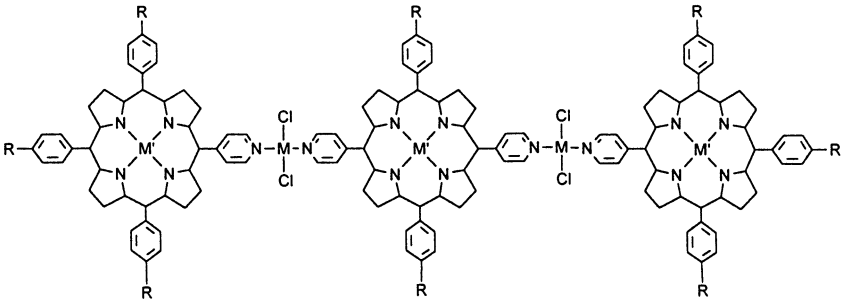

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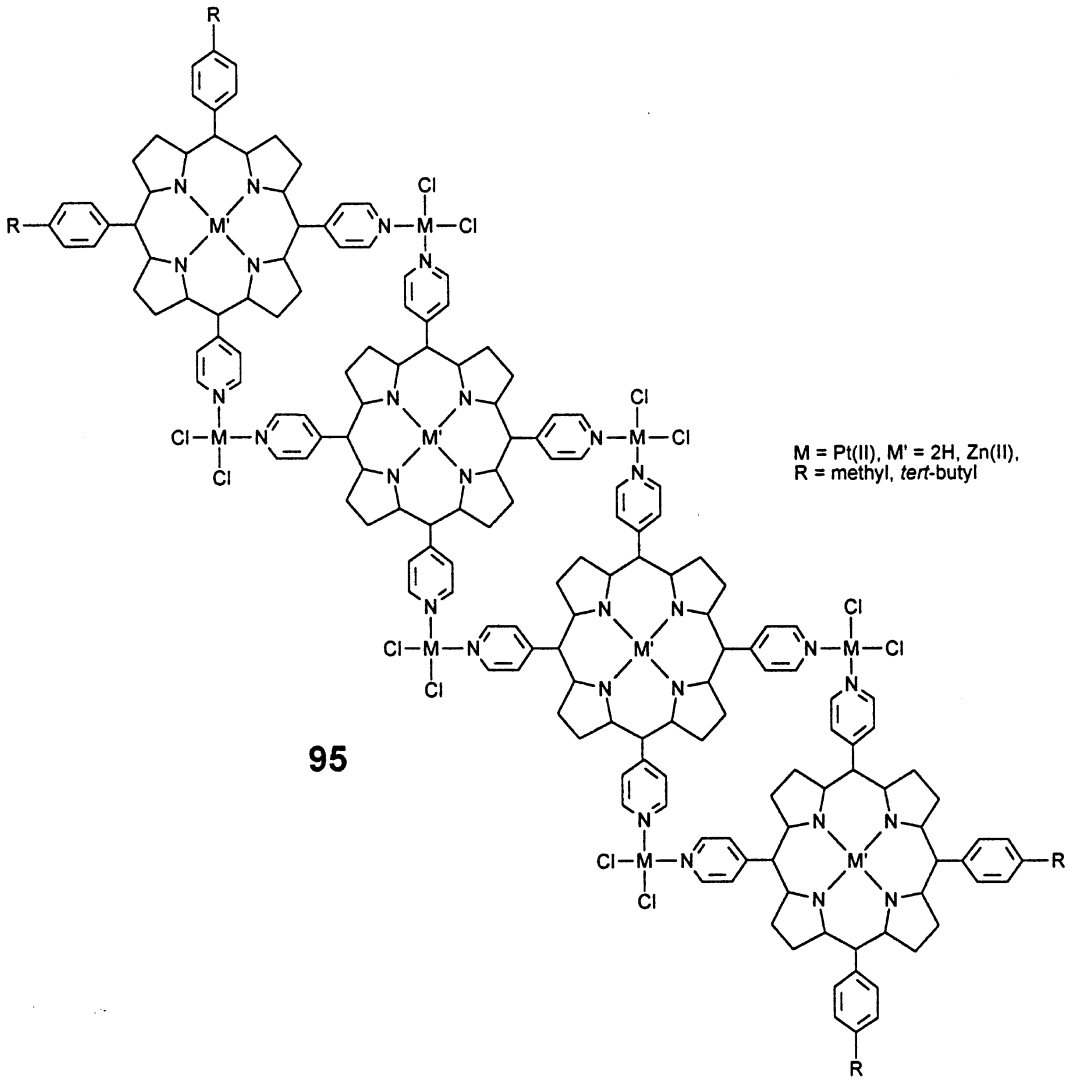

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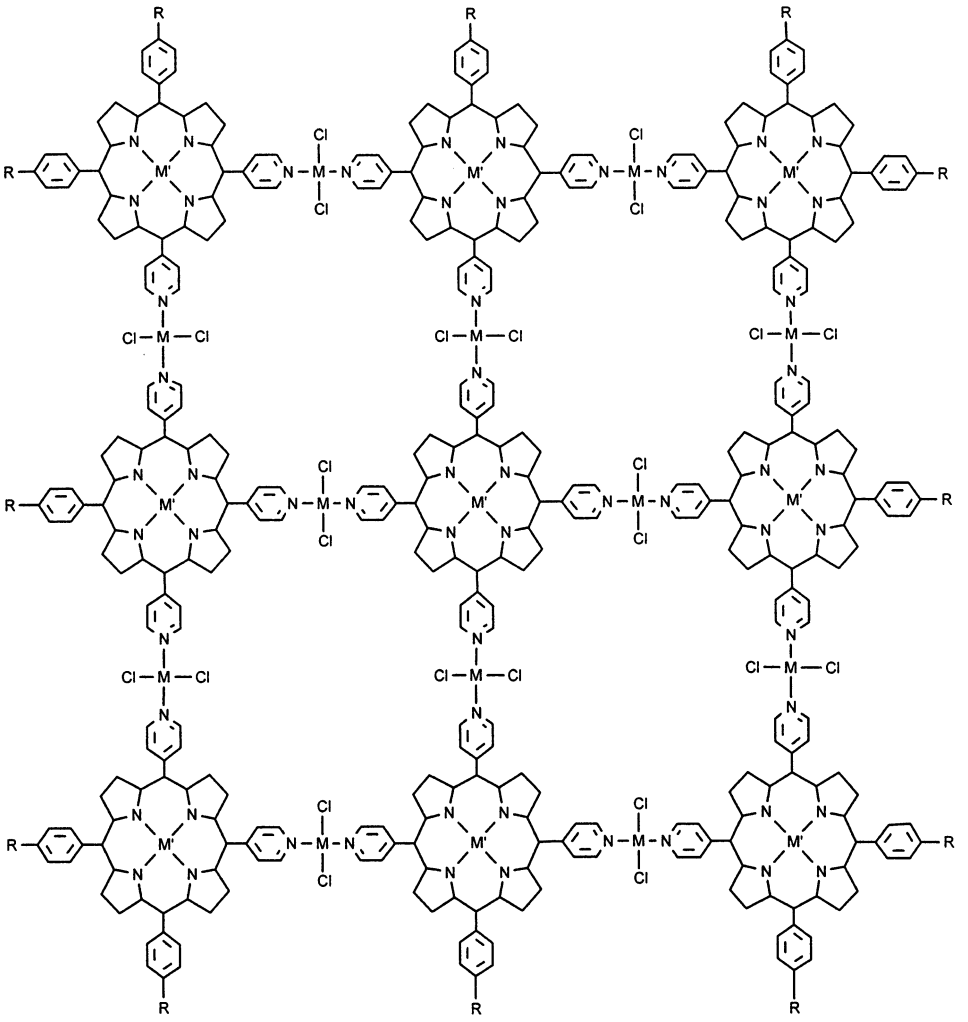
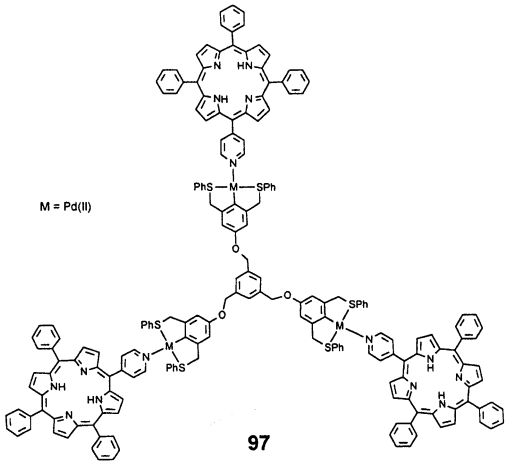


M = Pd(II), M' = 2H, Zn(II), R = methyl, *tert*-butyl

96



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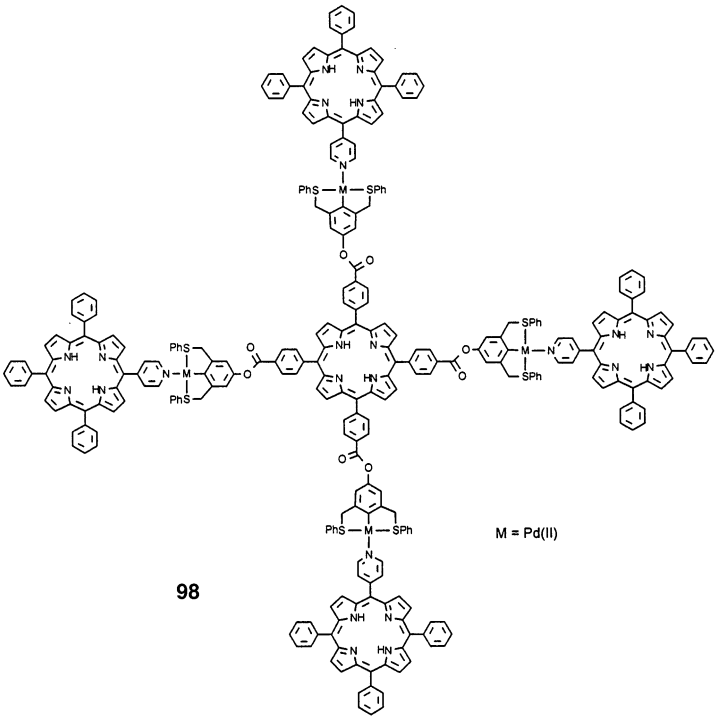


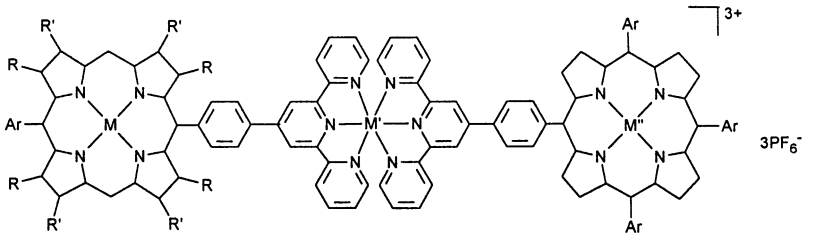
98

One-dimensional polymers formed by 5,10,15,20-tetrakis(4-pyridyl)porphyrins (and their zinc(II) complexes) interconnected by mercury(II) centers were recently reported [412,413]. Linking by cadmium(II) or lead(II) leads to a two-dimensional polymeric structure [413].

A new type of an infinite, three-dimensional polymeric network was constructed from complexes of tetrakis(4-pyridyl)porphyrin interconnected by Cd^{II}, Cu^{I} or Ru^{II} centers [414–416]. Copper(II) tetrakis(4-cyanophenyl)porphyrin building blocks were assembled into a network structure with large channels [415]. The square-planar building blocks were linked together by tetrahedral copper(I) coordinated to *meso*-(4-cyanophenyl) fragments.

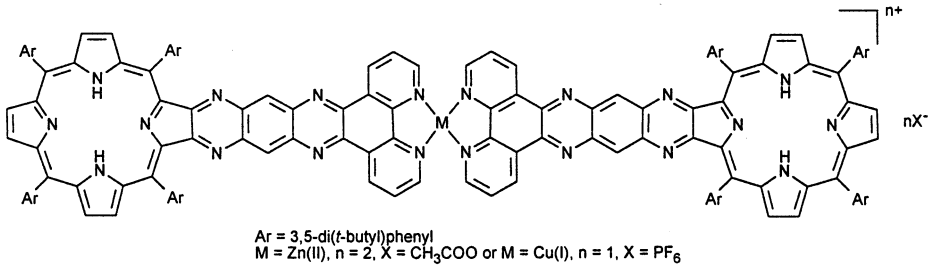
A self-assembled surface multilayer was reported that was based on *meso*-tetrakis(4-phosphonophenyl)porphyrin bound to zirconium(IV) [417].

Multicomponent systems consisting of two different metalloporphyrins and a central ruthenium(II) complex (e.g. **99** [418]) were obtained using the coordination of two porphyrin-substituted 2,2′,6′,2″-terpyridine ligands to the ruthenium ion [6,418–421].



99

The bis-phenanthroline complexes of copper(I) and zinc(II) were also used as spacers between two porphyrin fragments (e.g. in **100** [418], **101** [231]) [187,231,234–237,422–425]. Cu(I) ion was even used as a template for the synthesis of bis-porphyrin [2]catenates (e.g. **102** [422]) [235,422,424], a pseudorotaxane **103** [423] and a [2]rotaxane containing three porphyrin subunits **104** [234,236,237].



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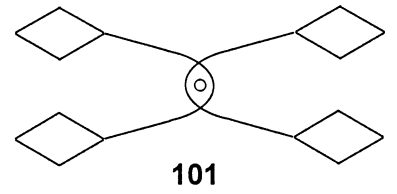

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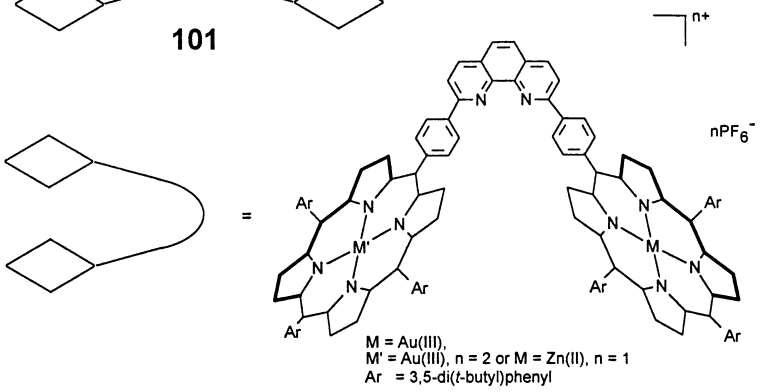
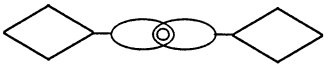


○ = Cu(I)



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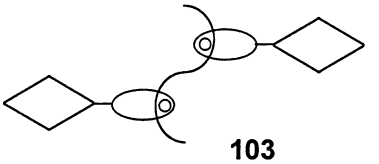
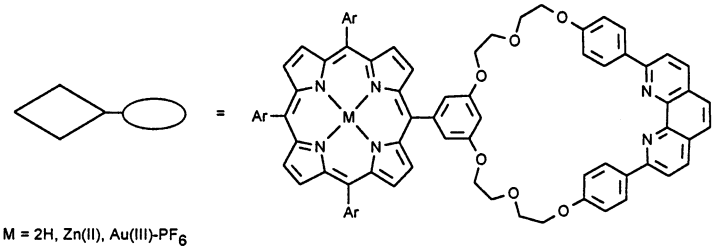
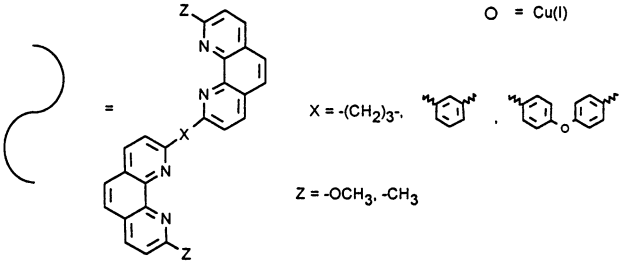
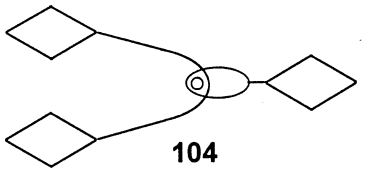


103

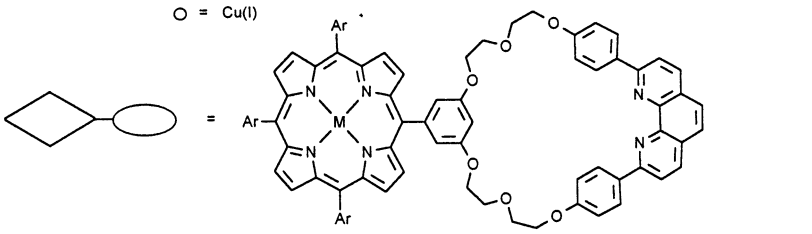


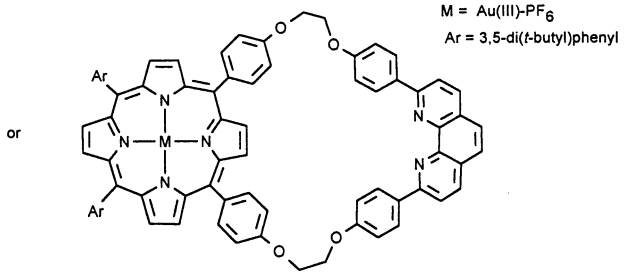


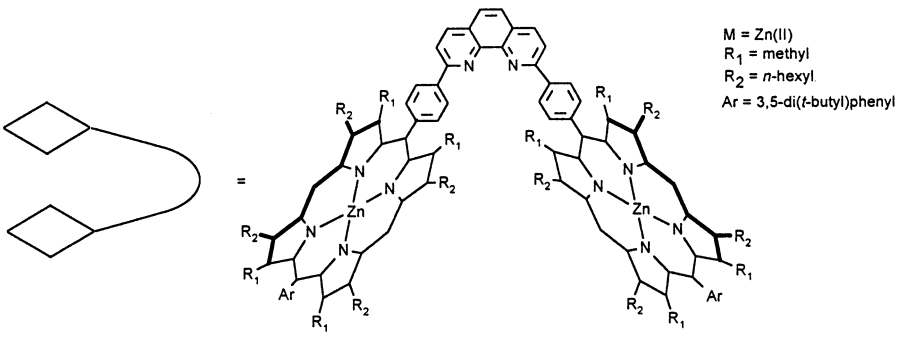
○ = Cu(I)



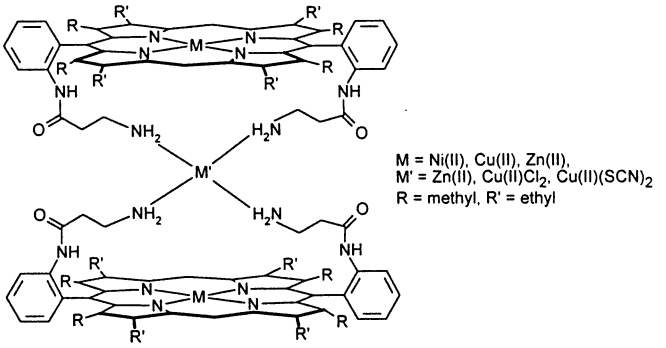
104







A binucleating ligand, *cis*-5,15-bis[*o*-(β-alanylamido)phenyl]-2,8,12,18-tetraethyl-3,7,13,17-tetramethyloporphyrin was used to obtain linear trinuclear metal complexes **105** where four appended amine groups of two porphyrins are coordinated to the bridging metal ion [426].

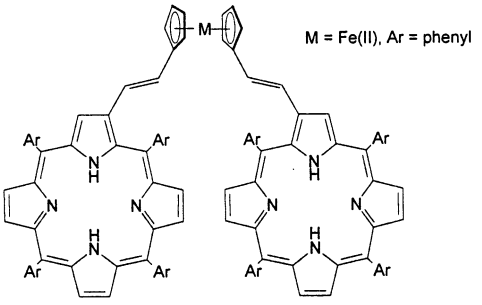


105

The K^+, Ba^{2+}, Cs^+, and NH_4^+ cations were found to induce dimerization of metalloporphyrins appended with crown ether molecules at four, three, or two adjacent *meso* positions [427–430]. Face-to-face dimers $(PM)_2M'_4$, $(PM)_2M'_3$, $(PM)_2M'_2$ (P = porphyrin, M = 2H, Mg(II), Ni(II), Cu(II), Zn(II), Mn(III), V(IV)O) are formed, respectively, in which M′ ions bring together pairs of crown ether rings of two neighboring porphyrinic units.

Similar dimers were observed in LSIMS and FAB mass spectra of tetraphenylporphyrins substituted with a crown ether in one phenyl ring, when Na^+ ions were present in matrix solution [431].

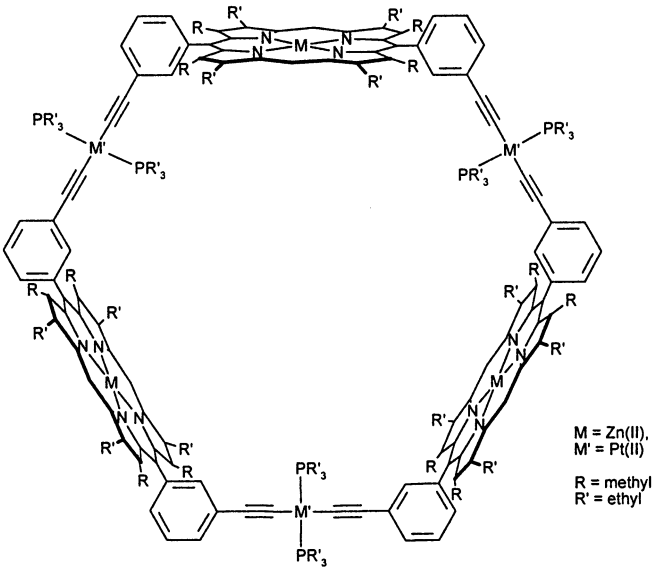
The dimeric porphyrins linked via ferrocene provide an interesting example of a diporphyrin system where an organometallic linkage was applied (e.g. in **106** [432]) [432,433].



106

Another type of organometallic motif was used for the construction of a cyclic

trimeric porphyrin **107** with alkyne–platinum–alkyne linkages [314,315]. Such a connection was also postulated in a series of linear zinc(II) porphyrin oligomers, that were detected by mass spectrometry [114].

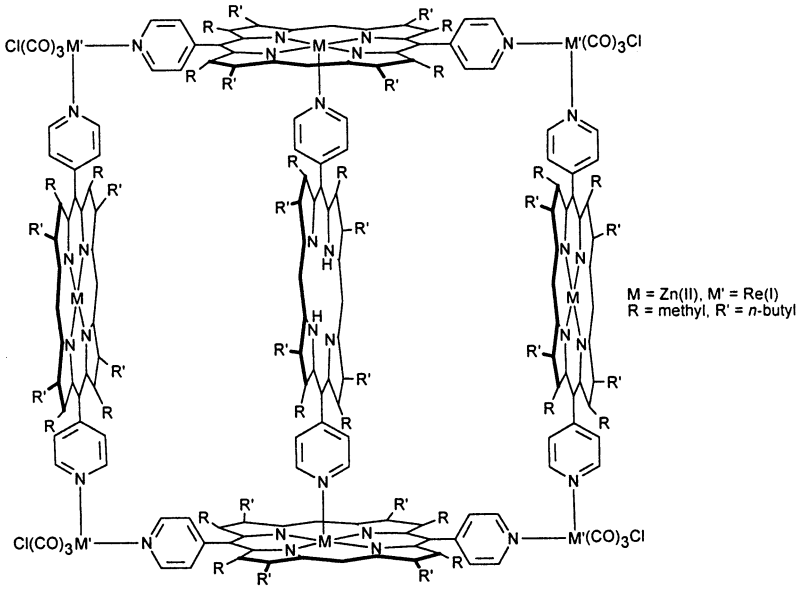


107

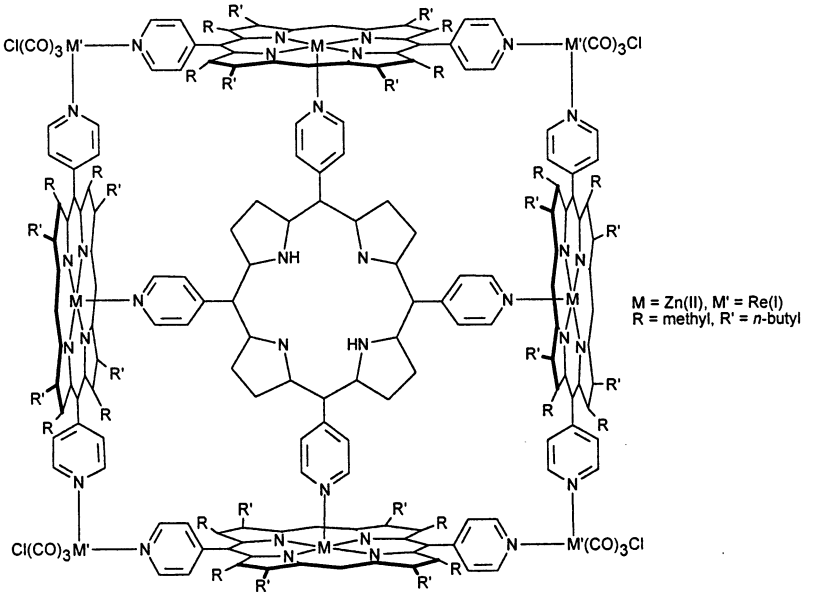
A two-dimensional network was obtained when *meso*-tetrapyridylporphyrins functionalized with four acetylenic groups were reacted with Ag(I) salts in Langmuir–Blodgett films [434]. Silver(I) diacetylide groups were found to link adjacent coplanar macrocycles.

In another organometallic assembly, two zinc(II) porphyrins were attached to a central Pt(II) ion via σ-alkynyl bonds [435].

Slone and Hupp prepared a molecular square based on four zinc(II) 5,15-bis(4-pyridyl)porphyrins and (chloro)*fac*-tricarbonylrhenium(I) corners [436]. They found that such a superstructure reveals the appropriate dimension and the multiple bonding site availability for strong host–guest interaction with 5,15-bis(4-pyridyl)porphyrin (complex **108**) and 5,10,15,20-tetrakis(4-pyridyl)porphyrin assembly (**109**). Interestingly two types of coordination linkage described in Sections 4.3 and 4.4 are simultaneously in use to construct these particular polymetalloporphyrin systems [436].



108



109

5. Conclusion

Significant developments in the construction of oligoporphyrins and oligometalloporphyrins have been seen over recent years. Considering the expansion of this field of chemistry, which continues to supply a large variety of new interesting compounds with potential use in electron and energy transfer, we have focused in this article on the specific role of the coordination linkage in creation of polyporphyrin architecture.

A supramolecular approach has been employed with great success to create a variety of porphyrin (metalloporphyrin) structures. Generally in this methodology, advantage is taken of the fact that metal centers play an essential structural and ordering role, which provides the requisite geometry in a controlled fashion.

In light of such a progress, one may anticipate a wide application of the self-assembly approach to prepare oligoporphyrins via coordination. To encourage the development of the inorganic aspects, the suitably designed porphyrins should be readily available using optimized, straightforward synthetic methods. The utility of the mixed 4-pyridyl/phenyl porphyrin as ubiquitous building block for the formation of a wide array of structures is quite enlightening in this respect. As a matter of fact these molecules set a stage for the further search for novel building blocks where the nature of the porphyrin and appended coordination centers will be appropriately adjusted.

In terms of future efforts in the area of oligometalloporphyrins, we expect that systems which include a more abundant assortment of metal ions and their complexes in the role of porphyrin building blocks and/or linking motifs will open novel routes of investigations. At present the domination of diamagnetic and inert metal ion complexes (palladium(II), platinum(II), ruthenium(II), zinc(II)) is clearly seen.

Thus, taking an advantage of the specific, previously created, architecture (e.g. dimers, discrete tapes, arrays or three-dimensional arrangements) new paramagnetic, redox active or catalytic centers can be spatially designed. In principle, magnetic interactions, electron transfer, cooperation of catalytic centers, and host–guest interactions of polymetallic and polyporphyrin assemblies should be tunable through the use of engineered architectures.

Acknowledgements

The financial support of the State Committee for Scientific Research KBN (Grant 3 T09A 155 15) is kindly acknowledged.

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