1
Introduction

In 1987, J.-M. Lehn, C.J. Pedersen, and D.J. Cram were honored with the Nobel Prize in Chemistry for their work on selective host–guest chemistry [1–3]. Since then, supramolecular chemistry has evolved into one of the most active fields within today's research community. This concept has been delineated by Lehn [4]:

> "supramolecular chemistry may be defined as 'chemistry beyond the molecule' and is based on organized entities of higher complexity that result from the association of two or more chemical species held together by intermolecular forces."

Self-recognition and self-assembly processes represent the basic operational components underpinning supramolecular chemistry in which interactions are mainly non-covalent in nature (e.g., van der Waals, hydrogen bonding, ionic or coordinative interactions). In general, these interactions are weaker and usually reversible when compared to traditional covalent bonds. Nature itself represents the ultimate benchmarks for the design of artificial supramolecular processes. Inter- and intramolecular non-covalent interactions are of major importance for most biological processes such as highly selective catalytic reactions and information storage [5]; different non-covalent interactions are present in proteins, giving them their specific structures. DNA represents one of the most famous natural examples, where self-recognition of the complementary base-pairs by hydrogen bonding leads to the self-assembly of the double helix. Starting with the development and design of crown ethers, spherands, and cryptands, modern supramolecular chemistry depicts the creation of well-defined structures via self-assembly processes [6] (similar to the well-known systems found in Nature [7]).

One of the most important interactions applied in supramolecular chemistry is metal-to-ligand coordination. In this arena, chelate complexes derived from N-heteroaromatic ligands, in particular based on 2,2′-bipyridine, 1,10-phenanthroline, and 2,2′:6′,2″-terpyridine (Figure 1.1), have become an ever-expanding synthetic and structural frontier.

Bipyridine has been known since 1888 when Blau first reported the formation of a bipyridine–iron complex [8]. One year later, Blau also synthesized and analyzed

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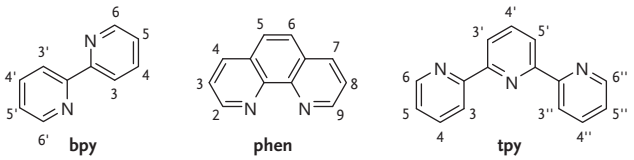

Figure 1.1 General structures of 2,2′-bipyridine (bpy), 1,10-phenanthroline (phen), and 2,2′:6′,2″-terpyridine (tpy).

bipyridine by dry distillation of copper picolinate [9]. Since this parent molecule consists of two identical parts, no directed coupling procedure is required for its construction. Therefore, unsubstituted and symmetrically substituted, in particular 4,4′-functionalized, bipyridines are readily accessible in good yields by simple coupling procedures [10, 11]. Apart from this, their transition metal (in particular Ru^{II}) complexes [12–14] feature interesting photochemical properties, making them ideal candidates for solar energy conversion, for example, in photovoltaic devices [15–23] and light-emitting electrochemical cells [24–28]. The chemistry of 2,2′:6′,2″-terpyridines (often referred to as simply terpyridine or tpy; the other structural isomers are duly noted but not considered further herein) is much younger than that of 2,2′-bipyridines. About 80 years ago, terpyridine was isolated for the first time by Morgan and Burstall by a process in which pyridine was heated (340 °C) in the presence of anhydrous $FeCl_3$ in an autoclave (50 atm) for 36 h [29, 30]; the parent terpyridine was isolated along with a myriad of other N-containing products. It was subsequently discovered that the addition of Fe^{II} ions to a solution of diverse terpyridines gave rise to a purple color indicative of metal complex formation.

Since this pioneering work, the chemistry of terpyridine remained merely a curiosity for nearly 60 years, at which point its unique properties were incorporated into the construction of supramolecular assemblies. Terpyridines and their structural analogs have gained much interest in the last two decades as functional templates in the fields of supramolecular and coordination chemistry as well as in materials science [31–38]. This is expressed by the enormous number of scientific publications and patents dealing with the synthesis, properties, and applications of terpyridine-containing systems (March 2011: about 5950 hits in SciFinder™, Figure 1.2). The terpyridine unit contains three nitrogen atoms and can, therefore, act as a tridentate ligand [39, 40]. The rich coordination chemistry and high binding affinity towards various interesting transition as well as rare earth metal ions, in concert with the resulting redox and photophysical properties, have given rise to diverse metallo-supramolecular architectures and a multitude of potential applications. Owing to their distinct photophysical, electrochemical, catalytic, and magnetic properties, terpyridines and their complexes have been studied regarding a wide range of potential applications covering light-into-electricity conversion [16, 41–60], light-emitting electrochemical cells (LECs) [61, 62], (electro)luminescent systems (e.g., organic light-emitting diodes) [63–68], and nonlinear optical devices [69–78]. Moreover, ditopic and dendritic terpyridine ligands may form

polymetallic species, which can then be utilized as luminescent or electrochemical sensors [79–134]. Besides these objectives, their biomedical and pharmaceutical utilizations (e.g., as DNA binding or antitumor active agents) are currently rapidly growing fields of research [79, 135–146].

Furthermore, the catalytic activity of terpyridines and their transition metal complexes has been employed to enhance various (asymmetric) organic transformations [147–149]: carbon–carbon single bond formation [150], etherification [151], oxidation of alcohols or ethers [152, 153], cyclopropanation [154, 155], epoxidation [156], Cu^{I}-catalyzed alkyne-azide cycloaddition (CCAAC) [157], hydrosilylation [158], and controlled radical polymerization to name only a few [159]. Additionally, Ru^{II} bis(terpyridine) complexes have also been used for the photocatalytic splitting of water [160–162].

Well-designed supramolecular (co)polymer architectures have been realized, based on the metal-terpyridine connectivity, opening up avenues to smart "self-healing" materials with the opportunity of switching the physical and/or chemical properties of materials depending on parameters such as pH value or temperature [36, 38, 163–173]. Finally, the self-assembly of terpyridine complexes onto nanostructures (e.g., based on gold, silver, CdS, TiO_2, carbon nanotubes) [174–178] as well as surfaces (e.g., glass, indium tin oxide, gold, graphite) [179–187] is considered in this context.

The diversity of applications related to terpyridines and their metal complexes calls for a high structural variability of the basic 2,2′:6′,2″-terpyridine subunit. In particular, terpyridine designs featuring π-conjugated substituents, commonly

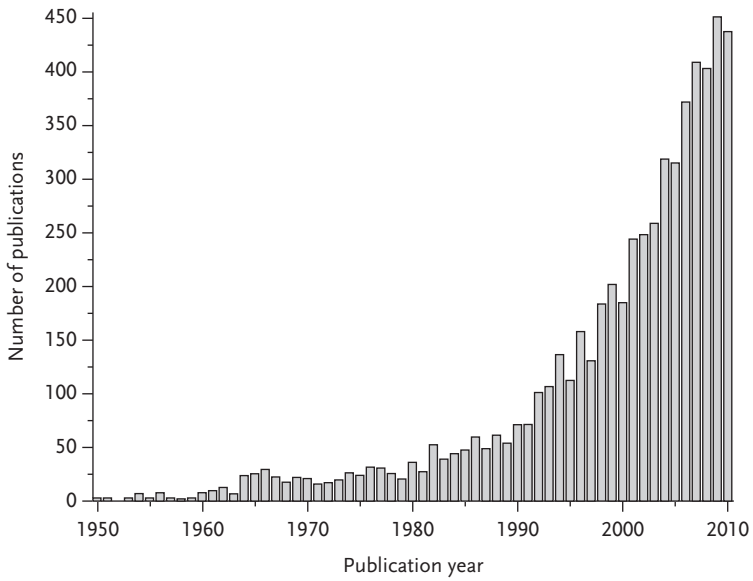

Figure 1.2 Histogram of the number of publications containing the term "terpyridine" using SciFinder™ (the search was performed on 1st March 2011).

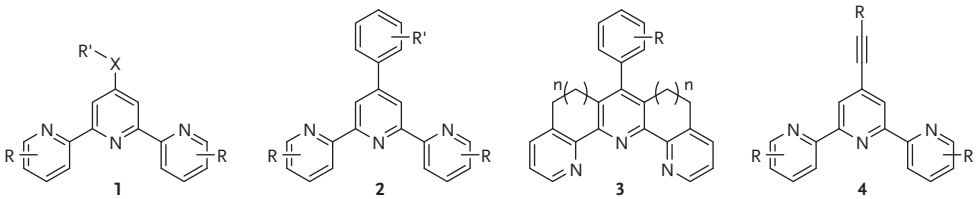

Figure 1.3 Chemical structures of 4′-functionalized (**1**, X = O, N or S), a Krǒhnke-type (**2**), a rigid U-shaped (**3**, $n = 0$, 1 or 2), and a Ziessel-type terpyridine (**4**).

attached in 4′-position, are of increasing interest. Figure 1.3 depicts the general schematic structures of four widely used types of terpyridines. Terpyridines (**1**) can be considered as "workhorses" in the field of metallo-supramolecular chemistry – a multitude of terpyridine-functionalized polymers has been derived from this structural motif [36, 38, 166, 171, 188, 189]. By far, most conjugated terpyridine-containing systems used today are based on the so-called Kröhnke-motif, which features a functionalized phenyl moiety at the 4′-position of the terpyridine unit (**2**) [37]. Their rigid U-shaped counterparts **3** have – mainly due to synthetic limitations – been employed less frequently [190] but offer entrée to a more rigid configuration. The Ziessel-type terpyridines **4**, where π-conjugation is extended via ethynyl-based systems, have been studied in particular with respect to electron-transfer processes [187, 191].

In view of the notable importance of 2,2′:6′,2″-terpyridines and their metal complexes in current research, we herein focus on architectures containing these types of ligand and their corresponding metal complexes.

The earlier book *Modern Terpyridine Chemistry* aimed mainly to summarize the syntheses, chemistry, and properties of functional terpyridine architectures: complexes, supramolecular polymers, 3D-structures, and surfaces [192]. Owing to the fast development of terpyridine-based materials, this book presents a detailed look beyond the basic concepts of syntheses and properties to applications with relevance to various aspects of human life. Therefore, this book consists of different topics related to "terpyridine-based materials," each of which is discussed in an individual chapter.

Chapter 2 summarizes the known synthetic strategies leading to different terpyridines. Since terpyridines of types **1**–**4** currently represent the most valuable derivatives, emphasis is laid on the discussion of the various routes of their syntheses. In this context, their properties, in particular their photophysical behavior, is also evaluated.

Chapter 3 describes the preparation and properties of mononuclear terpyridine metal complexes. Emphasis will be on bis(terpyridine) complexes of Ru^{II}, Os^{II}, Ir^{III}, and Pt^{II} ions as well as their photophysical and electrochemical properties. Moreover, oligonuclear complexes, such as dyads and triads, are included. In particular, architectures based on Ru^{II} ions are featured in which combinations with other transition metal ions could, for example, potentially lead to "molecular switches" opening up avenues to the construction of nanodevices.

Chapter 4 features more advanced supramolecular aggregates composed of terpyridine-metal subunits: macrocycles, grids, helicates, or rotaxanes. Such materials are of interest for the understanding of supramolecular aggregation into 2D and 3D architectures. Furthermore, applications as either "molecular machines" or optoelectronic devices have been envisioned.

The combination of π-conjugated bis(terpyridine)s with transition metal ions affords high molar mass π-conjugated metallopolymers; in these materials, the properties of conventional conjugated polymers and terpyridine complexes are merged (Chapter 5). Polymer light-emitting diodes (PLEDs) or polymer solar cells (PSCs) are the most prominent targets of research in this emerging field.

Polymeric architectures containing terpyridine systems with various architectures, from side-chain-functionalized polymers to main-chain metallopolymers, are summarized in Chapter 6. The incorporation of terpyridine complexes into polymer architectures enables the synthesis of advanced multiblock copolymers (that, for instance, can form micelles or phase-separate in the bulk) or polymer-bound photoactive metal complexes for optoelectronic applications.

Chapter 7 summarizes terpyridine metal complexes that have recently found application in the fields of biochemistry and pharmacy. In particular, Pt^{II} mono(terpyridine)s are potential cytotoxic agents that could be potential replacements for the traditional Pt^{II}-based drugs (e.g., cisplatin, carboplatin). Oxidative DNA cleavage, induced by various types of terpyridine complexes, is another major field in the biomedical arena. Photoluminescent complexes can be attached to biomolecules and, therewith, be utilized as labeling agents in pharmaceutical applications.

The covalent binding of terpyridines to surfaces has led to the development of molecular wires. Fast energy-transfer processes along these wires point to potential applications in organic electronics. Besides their attachment to surfaces, the binding of terpyridine ligands (or their complexes) to organic as well as inorganic nanomaterials will, then, be considered in Chapter 8.

Chapter 9 describes applications of terpyridines and their complexes in the fields of organometallic catalysis. Terpyridine ligands (and their complexes) have been used as homogeneous or heterogeneous catalysts in various types of (asymmetric) organic reactions; important contributions will be summarized. Utilization of photoactive terpyridine complexes in energy-transfer reactions will be considered with respect to "artificial photosynthesis" and photocatalytic water splitting reactions.

Finally, Chapter 10 provides a few concluding remarks.

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