

***N,N'*-CHELATING BIHETEROAROMATIC LIGANDS; A SURVEY**

EDWIN C. CONSTABLE

University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW (U.K.)

PETER J. STEEL

Chemistry Department, University of Canterbury, Christchurch (New Zealand)

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A. INTRODUCTION: PURPOSE AND SCOPE OF THE SURVEY

It is exactly 100 years since Blau first reported [1] the preparation of 2,2'-bipyridine (bpy) and described the first transition metal complex of this important ligand. Since then bpy and the related ligand 1,10-phenanthroline (phen) have been continuously and extensively used in both analytical and preparative coordination chemistry [2]. It is only relatively recently, however, that systematic studies of substituted derivatives of bpy and other α -diimine ligands have been undertaken. Much of this work has been initiated by the intense current interest in the redox and photocatalytic properties of the $\text{Ru}(\text{bpy})_3^{2+}$ cation [3].

Although substituents in the pyridine rings can significantly modify the physical and chemical properties of complexes containing bpy ligands, much greater changes can result from the replacement of one, or both, of the pyridine rings by other nitrogen-containing heterocycles. Such changes are explained by the very different electronic properties of the various nitrogen heterocycles [4]. Not only are there large variations in the σ -donating ability of the nitrogen donor atom but also there exist large differences in the π -acceptor/donor properties of the ligand. Most importantly the six-membered nitrogen heterocycles, being π -deficient, are excellent π -acceptors while the π -deficient five-membered heterocycles are much poorer π -acceptors and better π -donors. Such factors have important consequences in

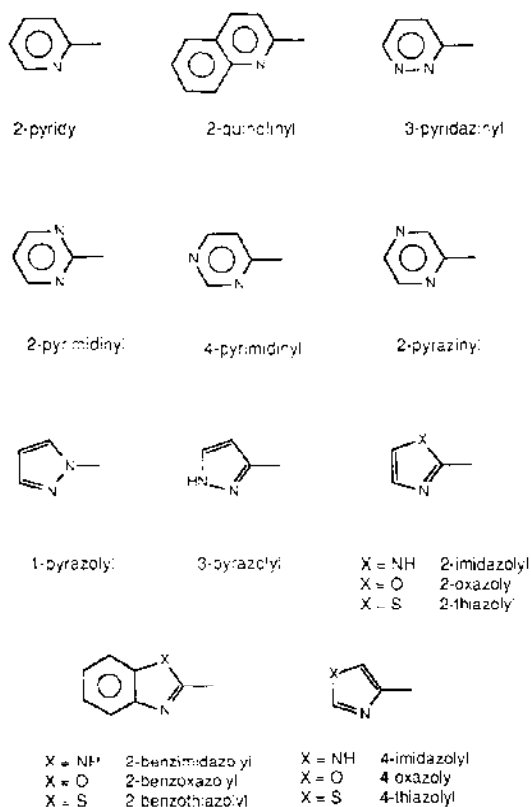


Fig. 1. Heterocyclic components of N,N' -chelating ligands.

attempts to tune the ground and metal-to-ligand charge transfer (MLCT) excited state properties of the $\text{Ru}(\text{bpy})_3^{2+}$ cation [3].

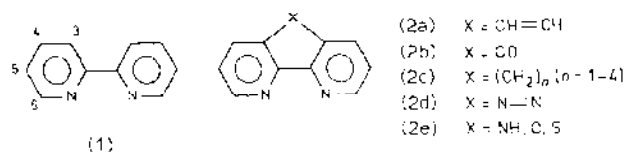
The purpose of the present report is to survey the known N,N' -chelating ligands which consist of two directly linked heterocycles from those shown in Fig. 1. Although the choice of heterocyclic components in Fig. 1 is somewhat arbitrary, this selection does represent the most commonly encountered heterocycles in coordination chemistry and represents a wide range of electronic properties for the ligands. Nevertheless, it has been found necessary to exclude some heterocycles (e.g. isoquinolines, triazoles) which are excellent ligands for transition metals. Since only aromatic heterocycles are considered, this also excludes the fully or partially saturated heterocycles such as imidazolines and thiazolines. Ligands formed by the direct linkage of two components from Fig. 1 form five-membered chelate rings on coordination to a metal. A number of other ligands exist which form

six-membered chelate rings and which consist of two heterocycles linked by a CH_2 , CO, NH, O or S group. Of these, dipyridylamine, dipyridyl ketone and the dipyrazolylmethanes are the most well known [2] but are also excluded from the present survey. Ligands consisting of one heterocyclic ring with a pendent nitrogen donor, such as an amine or imine nitrogen, are also not considered [2]. Furthermore, the discussion below is confined to N,N' -chelation of the ligands, some of which have been shown in recent years to be also capable of N,C' -chelation as cyclometallated complexes [2].

Of the 153 possible combinations of two components from Fig. 1, approximately one-half are presently known. However, only a small fraction of these has been extensively employed as ligands in coordination chemistry. It is hoped that the present survey will signal to coordination chemists the availability of many potentially important ligands the chemistry of which has yet to be explored. It is certain also that there is rich, untouched chemistry in the syntheses of the many remaining ligand combinations and in a systematic study of their coordination chemistry.

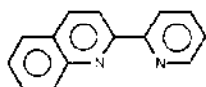
B. LIGANDS CONTAINING A PYRIDINE RING

The coordination chemistry of bpy (1) has been well reviewed elsewhere [2,5]. In the context of ruthenium(II) chemistry, many 3-, 4-, 5- and 6-substituted derivatives of bpy have been synthesized and their complexes prepared [3]. In addition to significant changes resulting from the electronic effects of substituents, substitution in the 6-position can impose severe steric constraints on coordination. Many 3,3'-bridged derivatives **2** are known of which phen (2a) and its substituted derivatives are the most well studied [6]. Others include the readily prepared diazafluorenone **2b** [7], the alkyl-bridged derivatives **2c** [8], the highly π -deficient aza-bridged ligand **2d** [9] and the heteroatom-bridged derivatives **2e** [10,11]. The coordination chemistry of **2b-2d** has recently been investigated. Chiral derivatives of bpy and phen have recently been synthesized for potential application in asymmetric catalysis [12,13].

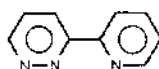


All but two of the 16 possible ligands formed by replacing one of the pyridine rings of bpy with another of the listed heterocycles are known. 2-(2'-Pyridyl)quinoline (**3**) has been much studied, with complexes of manganese [14], iron [15], ruthenium [16-18], osmium [18], copper [19] and gold [20] having been reported. As with other 6-substituted bipyridines,

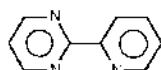
fusion of the benzo ring makes the ligand **3** sterically more demanding than bpy. X-ray structures of manganese and gold complexes have shown a significant elongation of the metal–nitrogen bond to the quinoline ring relative to that to the pyridine nitrogen [14,20]. In contrast to $\text{Fe}(\text{bpy})_3^{2+}$, which is low spin, the analogous complex of **3** is essentially high spin [15]. The additional electron delocalization and lowering of the ligand π^* orbitals from fusion of the benzo ring is manifested in the absorption spectra of the ruthenium and osmium complexes [18].



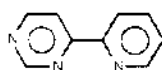
(3)



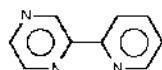
(4)



(5)



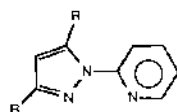
(6)



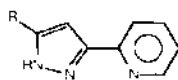
(7)

Although 3-(2'-pyridyl)pyridazine (**4**) itself is not known, 3,6-bis(2'-pyridyl)pyridazine has attracted much attention because of its ability to act as a binucleating ligand [21,22]. Very little work has been done with the other pyridyl diazines **5**–**7**. Reports describing the preparations of **5** and **6** make brief mention of the absorption spectra of their iron complexes [23,24], while ruthenium complexes of a methyl-substituted derivative of **5** have been reported [25,26]. The coordination chemistry of **7** is completely unexplored, in contrast to 2,3- and 2,5-bis(2'-pyridyl)pyrazine and 2,3,5,6-tetrakis(2'-pyridyl)pyrazine which have been well studied as binucleating ligands [27–32].

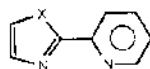
In recent years, there have been a number of reports of complexes of 2-(1'-pyrazolyl)pyridine (**8a**) and its dimethyl derivative **8b**. Complexes of **8a** with tin [33], mercury (including an X-ray structure) [34] and ruthenium [35] have been reported. ^{199}Hg NMR was used to demonstrate that the pyrazole ring is a better σ -donor for mercury than is pyridine [36]. Complexes of **8b** with nickel, cobalt, copper [37], ruthenium [38], palladium, platinum [39],



(**8a**) R = H
(**8b**) R = Me



(**9a**) R = H
(**9b**) R = Me
(**9c**) R = Ph

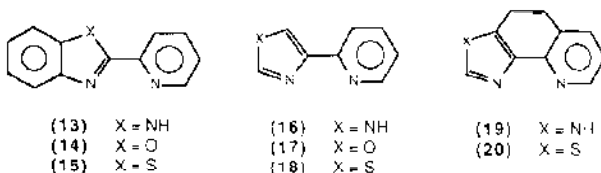


(**10**) X = NH
(**11**) X = C
(**12**) X = S

gold [40], tin [41] and uranium [42] have all been described. Mixed-ligand Ru^{2+} complexes of **8a** and bpy have been studied, from which it was deduced that **8a** is a much weaker π -acceptor than bpy [38]. Although the unsubstituted 3-pyrazolyl ligand **9a** is not known, the methyl and phenyl derivatives **9b** and **9c** have been reacted with a variety of metal ions [43,44] including ruthenium [45]. These ligands can also act as anionic bidentate ligands after deprotonation of the (non-coordinating) N-H nitrogen and both (neutral and anionic) modes of coordination are known [43].

There have been numerous studies of complexes of various metals with 2-(2'-imidazolyl)pyridine (**10**), which can also act as both a neutral and anionic ligand [46–53]. Much of the interest in this ligand has been generated by the unusual magnetic properties of the Fe^{2+} complex of **10** which exhibits spin cross-over behaviour in the protonated form only [48,52,53]. From a study of mixed-ligand Ru^{2+} complexes of **10**, it was concluded that the deprotonated form of **10** is a stronger π -donor than the protonated form which in turn is stronger than bpy [50]. Although the 2-oxazolyl analogue **11** has been known for some time [54], the first coordination chemistry of this ligand has only recently been reported [55]. In contrast, the 2-thiazolyl ligand **12** has been much studied, with complexes of iron, cobalt, nickel, copper, zinc and ruthenium having been described [55–58]. Interestingly, this ligand has similar π -acceptor properties to bpy, which has been attributed to back-bonding into the vacant d orbitals of the sulphur [57]. No spin cross-over behaviour was observed for the Fe^{2+} complex of the type mentioned above for the analogous complex of **10**.

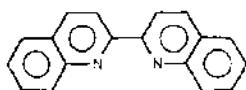
The three benzo fused analogues **13–15** are all known. Being commercially available, the benzimidazole **13** has been much studied as a ligand for numerous transition metals [40,48–50,59–61]. The Fe^{2+} complex exhibits magnetic properties similar to those described above for **10**. From studies of Ru^{2+} complexes of **13**, it was concluded [50] that this ligand has π -donor properties intermediate between those of bpy and the imidazole **10**. The benzoxazole **14** has not been used as a ligand, although complexes with numerous metals have been reported for a methyl-substituted derivative [62–65]. From studies of nickel and iron complexes of the benzothiazole **15**, it was concluded [66] that this is a weaker ligand than the corresponding thiazole **12**. Again numerous complexes of a methyl-substituted derivative have been prepared [62–65].



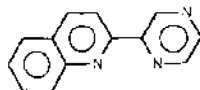
Although the 4-imidazolyl ligand **16** has been shown [67] to form more stable complexes than the 2-imidazolyl isomer **10**, there are only two early reports of its use as a ligand for five first row transition metals [67,68]. It was shown that this ligand is a weaker π -acceptor than bpy. The corresponding oxazole **17** is not known, while the thiazole **18**, like its isomer **12**, has been shown [69] to have similar electronic properties to bpy. X-ray crystal structures of nickel and iron complexes of **18** have been determined [69]. 3,5'-Bridged derivatives of **16** and **18** are known. Complexes of ligands containing the imidazoloquinoline moiety **19** have been reported [43], while the triazolo analogue **20**, although known for almost 50 years [70], has not been employed as a ligand.

C. LIGANDS CONTAINING ANOTHER AZINE

A number of ligands are known which contain the 2-quinolinyl moiety. Of these the commercially available ligand 2,2'-biquinoline (**21**) is by far the most well studied and has been complexed with numerous transition metal ions [15,71-80]. This is a very bulky ligand, as is exemplified by its reluctance to form tris complexes with Fe^{2+} [15,72] and by the lability of such complexes. Several X-ray structures of complexes containing **21** have been determined [73-75] and detailed spectroscopic and electrochemical studies of ruthenium complexes reported [79,80]. Ruthenium complexes of several 3,3'-bridged derivatives of **21** have been described [81] and metal complexes of a derivative of **21** possessing additional pendent nitrogen donors have recently been reported [82].



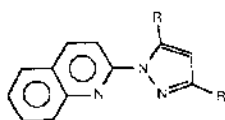
(21)



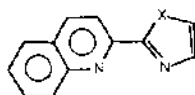
(22)

Of the four possible diazinyloquinolines, only the 2-pyrazinyl derivative **22** is known [83], although a trisubstituted derivative of 2-(2'-pyrimidinyl)quinoline has been prepared [84]. The coordination chemistry of such compounds is completely unexplored.

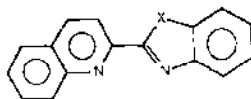
Although 2-(2'-pyrazolyl)quinoline (**23a**) is not known, several substituted derivatives have been prepared [85,86]. Iron, cobalt, nickel and copper complexes of the sterically hindered dimethyl derivative **23b** have been prepared and the X-ray structure of a cobalt complex determined [87]. All three 2,2'-substituted ligands **24-26** are known [88-90], yet none has been used as ligands in coordination chemistry. Of the benzo-fused derivatives **27-29**, the benzimidazole **27** has been complexed with iron, cobalt, nickel



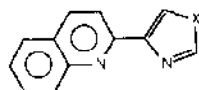
(23a) R = H
(23b) R = Me



(24) X = NH
(25) X = O
(26) X = S



(27) X = NH
(28) X = O
(29) X = S

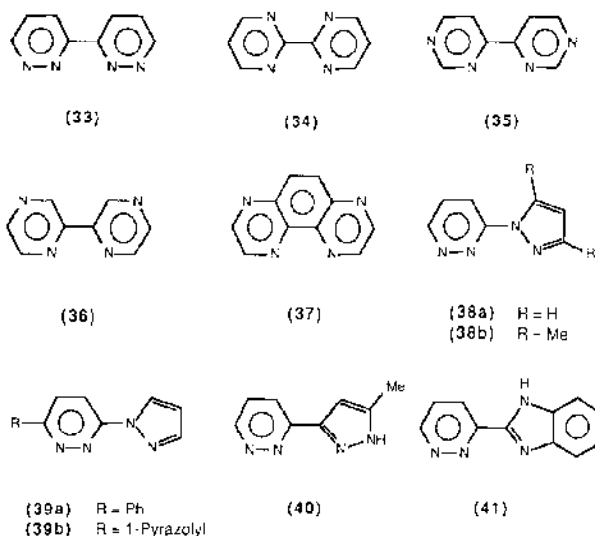


(30) X = NH
(31) X = O
(32) X = S

and copper [91–93]. Substituted derivatives of **27–29** have also been reacted with various transition metals [62–65,93]. Of the 2,4'-linked ligands **30–32**, only the imidazole **30** is known [94], although derivatives of **32** possessing additional substituents have been prepared [95,96]. Again no coordination chemistry of these compounds has been reported.

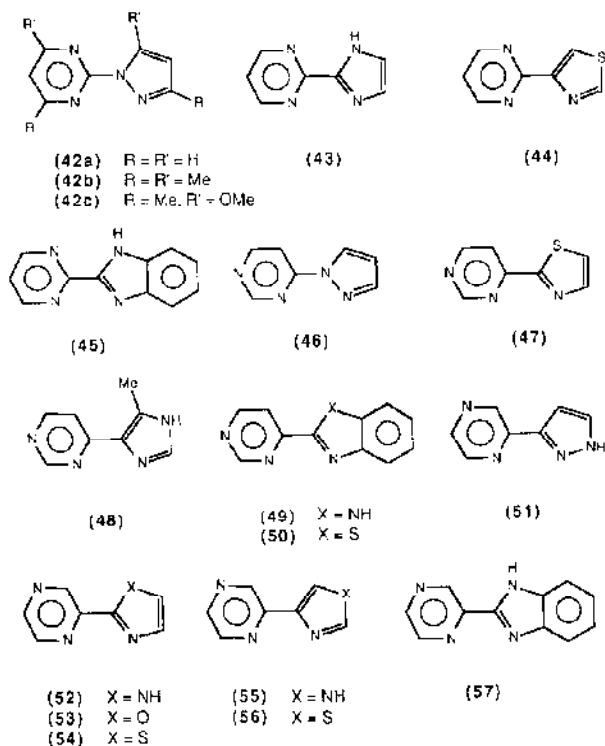
Of the ten possible chelating bidiazines, only the four symmetrically coupled isomers **33–36** are known. Although weaker σ -donors than bpy, these ligands are all stronger π -acceptors. Indeed the relative π -acceptor properties of these ligands have been determined by spectroscopic studies of their complexes with various d^6 and d^8 transition metal ions [97–100], and the spectroscopic and electrochemical properties correlated with LUMO energies derived from extended Hückel molecular orbital calculations [80]. The ligands **34** and **36** are both now commercially available, the former having been extensively studied because of its ability to act as a bis-bidentate binucleating ligand for the formation of both homobimetallic and heterobimetallic complexes [27,97,101–105]. 2,2'-Bipyrazine (**36**) has attracted considerable attention in recent years [3] principally owing to reports that its ruthenium complex is a superior photocatalyst to $\text{Ru}(\text{bpy})_3^{2+}$. Ruthenium complexes of the 3,3'-bridged derivative **37** have also been reported [106].

Ligands formed by coupling a diazine to an azole are less common and their coordination chemistry has not been systematically studied. Only a few pyridazine-containing ligands are known. Although the parent 1-pyrazolyl ligand **38a** has been prepared [107], the only coordination chemistry re-



ported is of substituted derivatives. Rhodium complexes of the dimethyl derivative **38b** have been reported [108] and a palladium complex prepared [109] from the phenyl-substituted derivative **39a**. The related dipyrazolylpyridazine **39b** has been employed as a binucleating ligand for copper [110] and several X-ray structures of such complexes have recently been reported [111,112]. The 3-pyrazolyl compound **40** and the benzimidazole **41** are also known [113,114].

Numerous substituted derivatives of 2-(1'-pyrazolyl)pyrimidine **42a** have been synthesized for pharmaceutical evaluation [115–117]. Cobalt, nickel and copper complexes of the tetramethyl derivative **42b** have been prepared [118], while there have been several reports [119–123] of complexes of the important anti-inflammatory agent mepirizole **42c**, including two X-ray structure determinations of copper complexes [119,123]. Some substituted derivatives of the 2-imidazolyl- and 4-thiazolyl-pyrimidines **43** and **44** are also known [124,125]. The benzimidazole **45**, which could potentially act as a monoanionic binucleating ligand, has also been prepared [114]. Many substituted derivatives of 4-(1'-pyrazolyl)pyrimidine (**46**) are known [115–117,126] and rhodium complexes of 4,6-bis(3,5-dimethyl-1-pyrazolyl)pyrimidine have been reported [108]. The 4-substituted pyrimidines **47–50** are all known [127–129] but have yet to be employed as ligands. Substituted derivatives of the pyrazines **51–56** are known [130–132] but the only coordination chemistry of such compounds which has been published is



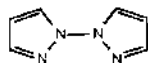
a very recent report [58] of a ruthenium complex of the thiazole **54** in which detailed 1H and ^{13}C NMR studies of the RuL_3^{2+} complex led to full spectral assignments of both the meridional and facial isomers. The benzimidazole **57** has also been prepared as a bpy analogue [114,133].

D. LIGANDS CONTAINING TWO AZOLES

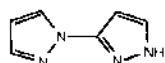
Of the 66 ligands possible from coupling two of the azole components in Fig. 1, almost one-half are known. Although the majority of these have been synthesized for the screening of their biochemical activities, it is nevertheless surprising that only four of these have been commonly used as ligands in coordination chemistry. In view of the interesting chemistry that has been reported for these ligands in recent years, it is likely that future studies of related ligands will provide equally interesting results.

Of the three possible bipyrazoles **58–60** the *N,N'*-linked isomer **58** has not yet been prepared, although substituted derivatives have recently been

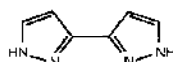
reported [134]. The *N,C'*- and *C,C'*-linked isomers **59** and **60** are both known [135,136] but complexes of these ligands have yet to be reported. However, all three isomers **58–60** have been incorporated into metal-complexing macrocyclic ligands [137–139].



(58)



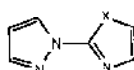
(59)



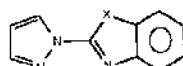
(60)

Although the 2-substituted imidazole **61** has been described as unstable towards hydrolysis [140], some substituted derivatives have recently been reported [141]. Numerous substituted derivatives of the thiazole **62** and the benzazoles **63–65** have been prepared and screened for biological activity [142–152]. The 3-pyrazolyl compounds **66** and **67** and substituted derivatives of **68** are all known [153–155], as are the 2-(3'-pyrazolyl)benzazoles **69** and **70** [156,157]. However, the coordination chemistry of all these potentially interesting ligands (**61–70**) remains unexplored.

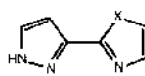
In contrast, 2,2'-biimidazole (**71**) has been extensively used as a ligand for many years. This readily prepared [158,159] ligand can act in a neutral bidentate mode or, after deprotonation, as an anionic or dianionic ligand. Furthermore, the doubly deprotonated form can act as a bridging bis-bidentate ligand in binuclear complexes. All these forms of coordination have been reported and complexes with numerous metals described [51,102,159–163]. X-ray crystal structures of the free ligand [164] and several mono-nuclear and both homobinuclear and heterobinuclear metal complexes have



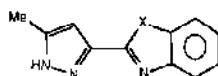
(61) X = NH
(62) X = S



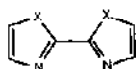
(63) X = NH
(64) X = C
(65) X = S



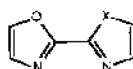
(66) X = NH
(67) X = NMe
(68) X = S



(69) X = NH
(70) X = S



(71) X = NH
(72) X = O
(73) X = S

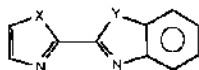


(74a) X = NH
(74b) X = S

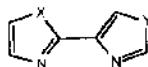
been reported [163–170]. Studies have shown that the acidity of the NH protons increases substantially on coordination and that, as expected, the ligand is a stronger π -donor than the imidazolylpyridine **10** or bpy [171]. Complexes of the tetramethyl and the π -deficient tetracyano derivatives of **71** have also been described [172–176]. Other substituted derivatives of **71** have been prepared, including *N,N'*-bridged derivatives [177–178].

The only coordination chemistry reported for the bioxazole **72** or bithiazole **73** is in very early descriptions [179,180] of iron, nickel and copper complexes of **73** and in very recent reports of its complexes with ruthenium [55,58]. A number of aryl-substituted derivatives of **72** and **73** have been prepared [181–183] and an X-ray structure of a dimethyl derivative of **73** reported [184]. The partially saturated 4,4',5,5'-tetrahydro derivatives of **71–73** have also frequently been employed as ligands in recent years [185]. The unsymmetrical 2,2'-biazole **74a**, its benzothiazole analogue and the substituted biazole **74b** have recently been prepared [90,186].

The three monobenzo 2,2'-biazoles **75–77** are all known [90,187], as are a number of substituted derivatives [188,189]. Given the current interest in complexes of 2,2'-biimidazole (**71**) and bibenzimidazole (see below), it is surprising that the related ligand **75** has not been similarly investigated. Of the 2,4'-biazoles, substituted derivatives of **78** and **79** and the parent ligands **80** and **81** have all been prepared [190–194].



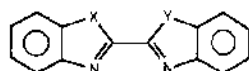
- (75) X = Y = NH
(76) X = S, Y = NH
(77) X = Y = S



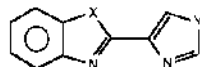
- (78) X = Y = NH
(79) X = NH, Y = S
(80) X = S, Y = O
(81) X = Y = S

All six 2,2'-bibenzazoles **82–87** have been known for some time and are available from several different synthetic routes [187,195–200]. Curiously, however, only 2,2'-bibenzimidazole (**82**) has been used as a ligand for coordination to metal ions. Like 2,2'-biimidazole (**71**), this ligand can coordinate in a variety of modes and both mononuclear and binuclear complexes with numerous transition metals have been reported [159,160,168,171,201–205]. An important recent paper [210] described detailed spectroscopic and electrochemical properties of homobimetallic and heterobimetallic ruthenium, osmium, cobalt and nickel complexes bridged by the dianion of **82**. In combination with molecular orbital calculations, it was shown that this ligand is both a strong σ -donor and π -donor and that these complexes are considerably more stable than analogous complexes

bridged by 2,2'-bipyrimidine (**34**). The greater stability of the 2,2'-bibenzimidazole-bridged complexes was attributed to a combination of the superior donor properties of the ligand and a reduction in electrostatic repulsion between the two positively charged metal centres when bridged by the dianionic ligand [201].



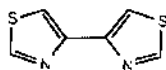
- (82) X = Y = NH
 (83) X = Y = O
 (84) X = Y = S
 (85) X = NH, Y = O
 (86) X = NH, Y = S
 (87) X = O, Y = S



- (88) X = NH, Y = NMe
 (89) X = Y = O
 (90) X = Y = S
 (91) X = S, Y = NPh
 (92) X = NH, Y = S

The 2,4'-linked compounds **88–91** are all known but have not yet been used as ligands [206–209]. In contrast, thiabendazole (**92**), which is a widely used anthelmintic and fungistatic agent, has been extensively studied and complexes with numerous metals reported [210–217]. X-ray crystal structures of the free ligand **92** [218] and of cobalt [211] and copper [214] complexes have been determined. The ligand generally acts in an *N,N'*-chelating mode, although one recent report [217] has suggested that in a complex with mercuric chloride the ligand is π -bonded through the imidazole ring.

Of the 4,4'-biazoles, only the symmetrical 4,4'-bithiazole (**93**) has been studied. Although iron, nickel, copper and platinum complexes of **93** were described almost half a century ago [179,180], this ligand was subsequently ignored until relatively recently when a mixed-ligand ruthenium complex containing **93** was reported [219]. In connection with magnetic studies, X-ray crystal structures of iron and nickel complexes of **93** have been reported [215]. Substituted derivatives of **93** are also known [220].



(93)

F. APPENDIX: LIGAND SYNTHESSES

The following list gives references to the best and most convenient method for preparation of the ligands discussed above: **2b** [7], **2c** [8], **2d**

[10,11], **3** [15,20], **5** [23], **6** [221], **7** [222], **8a** [223], **8b** [38], **9b** [44], **9c** [224], **10** [48], **11** [54], **12** [225], **14** [199], **15** [226], **16** [227], **18** [228], **20** [70], **22** [83], **23b** [85], **24** [88], **25** [89], **26** [90], **27** [229], **28** [199], **29** [230], **30** [94], **33** [23], **35** [231], **37** [232], **38a** [107], **38b** [108], **39a** [109], **39b** [233], **40** [113], **41** [114], **42b** [118], **42c** [234], **45** [114], **47** [127], **48** [128], **49** [129], **50** [129], **54** [58], **57** [114], **59** [135], **60** [136,229,235], **66** [153], **67** [154], **69** [156], **70** [157], **71** [158,159], **73** [193,236], **74a** [90], **74b** [186], **75** [187], **76** [187], **77** [90], **80** [192], **81** [193,194], **82** [159], **83** [197], **84** [196,200], **85** [187], **86** [198,199], **87** [198,199], **88** [206], **89** [207], **90** [208], **91** [209], **92** [237], **93** [193,238].

Ligands **1**, **2a**, **13**, **21**, **34**, **36** and **92** are commercially available.

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