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Rational design of metal coordination compounds with azomethine ligands

A D Garnovskii, I S Vasil'chenko

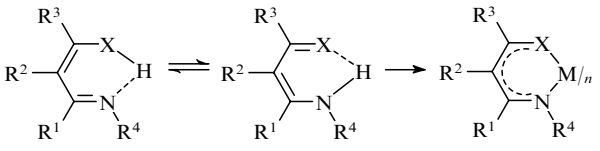
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Abstract. This review surveys the state of art in the coordination chemistry of chelating azomethine systems, *viz*., amino(hydroxy)-azomethines, β-aminovinyl ketones, β-aminovinylimines and their sulfur- and selenium-containing analogues. Variations in the fine structure of azomethine ligands allow one to perform the targeted synthesis of chelate and molecular, mono-, bi- and polynuclear, homo- and heterometallic structures. The bibliography includes 425 references.

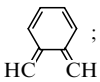
I. Introduction

Advances in coordination chemistry are to a large extent associated with the construction of new types of ligand systems. However, there are some well-known rather simple ligands, which can also allow progress in the chemistry of complex compounds relying on the principles of rational design (variation in the fine structure of ligand systems). Among these ligands are, for example, Schiff bases (azomethines) and their structural analogues, which are the 'oldest' [1,2] and most studied [3–24] ligands in coordination chemistry. The complexes with these ligands have attracted interest for more than 150 years primarily because the nature of substituents in azomethine ligand systems **1** can be varied over a wide range and it is possible to prepare mononuclear metal chelates **2** exhibiting various physicochemical properties and containing coordination units with different geometry.



1a **1b** **2**

R^1, R^2, R^3, R^4 = H, Alk, Ar, HetAr; $R^2 - R^3$ = CH=CH–CH=CH,



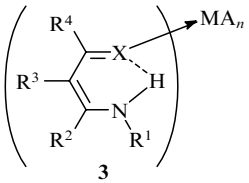
; X = NR^5, O, S, Se; R^5 = H, Alk, Ar, Ts; Ts = $SO_2C_6H_4Me$-4.

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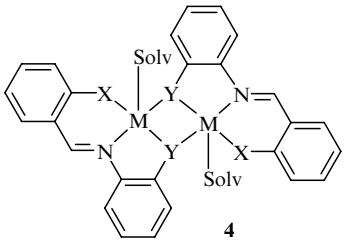
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In earlier reviews [2–23] and numerous more recent papers, most attention has been concentrated on azomethine-based chelate compounds. However, not only chelates **2** but also other structural types of metal complexes of chelate-forming azomethines have been much studied.[20,22,24–33]

Taking into account the classification proposed by one of the authors of the present review,[24–26] complex compounds of Schiff bases and their analogues can be divided into the following three types: chelates **2**; molecular complexes **3**; bi- and polynuclear compounds (**4** and **5**) and polymeric structures (**6**).

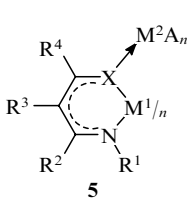


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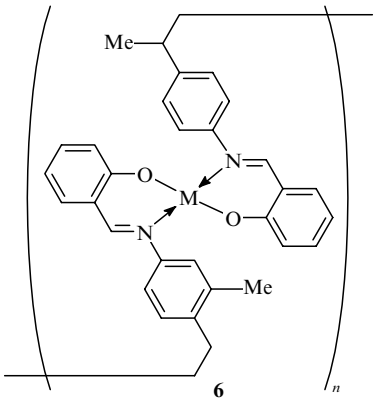


4

$R^1 - R^4$ = Alk, Ar; X, Y = NTs, O, S; M = Ni, Cu, Zn, A = Hal, NO_3; M = B, Al, Sn, Ti, Zr, Hf, Th, Zn, Cd, Hg; n = 2–4.



5

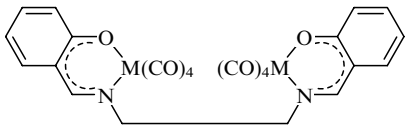


6

M = Co, Ni, Cu.

All attempts to synthesise the fourth type of complexes, *viz*., π-complexes,[24–26] with azomethine ligands failed because, apparently, of a shortage of complex-forming agents and the lack of procedures for the synthesis, which could be used for the preparation of these (predominantly organometallic [24,34–36]) compounds. However, it should be noted that the reactions of *N*,*N*′-bis(salicylidene)ethylenediamine with typical π-complex-forming

agents, *viz*., metal hexacarbonyls, afforded chelate compounds **7**.[34]



M = Cr, W. **7**

Metal chelates involve deprotonated (anionic) di- or polydentately coordinated azomethine ligand systems. Neutral chelate-forming azomethine compounds **3**, most of which are monodentately coordinated to the metal atom, serve as ligands in molecular complexes. Chelates **4** as well as compounds **5**, which are adducts of chelate compounds **2** and Lewis acids (M^2A_n),[24, 30] and metal-containing azomethine polymers **6**[31–33] can be assigned to bi- and polynuclear structures.

The present review surveys the data (published predominantly in the last decade) on the synthesis and structures of different types of azomethine metal complexes. Particular attention is given to the choice and modifications of ligand systems (rational design) and procedures for the synthesis of complexes of Schiff bases and their analogues, which are of importance in the preparation of compounds with the desired mode of arrangement of the coordination bond and geometry of the metallocycles as well as in the construction of complexes of different nuclearity and with different degrees of oligomerisation.

II. Azomethine metal chelates

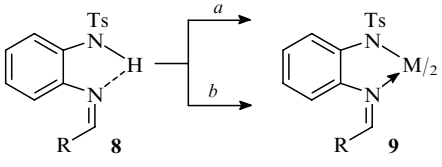
This section is devoted primarily to the employment of azomethine compounds, which have an intramolecular hydrogen bond giving rise to a five- or six-membered coordination unit and contain varying aldehyde and amine fragments, as chelating ligands. Chelate compounds based on these ligands can be prepared by reactions of ligand systems with metal salts as well as with the use of template and electrochemical synthesis methods.[24]

1. Chelate compounds with five-membered metallocycles

a. Complexes with N,N-donor atoms

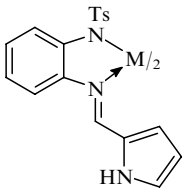
Aromatic azomethine chelates containing the five-membered MN_4 coordination unit became available in 1977.[37] These compounds are prepared by the reactions of Schiff bases of monotosylated *o*-phenylenediamine with metal acetates (method *a*)[17, 37–39] or metals under the conditions of electrosynthesis. The latter method involves the reactions of zero-valent metals (anode) with ligands in an electrolytic cell equipped with a platinum cathode (method *b*).[39–43]

Compounds **8** contain aromatic[37, 38] and heterocyclic[38–43] fragments as the substituent R.

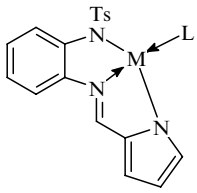


R = Ar, HetAr; M are 3*d* metals.
(*a*) $M(OAc)_2$, MeOH, Me_2CO; (*b*) M^0, $-2e^-$, MeCN.

In compounds **9**, ligands containing the pyrrole substituents, which are capable of forming additional coordination bonds, can serve as both bidentate ligand systems **10**[40, 41] and tridentate ligand systems **11**.[41]

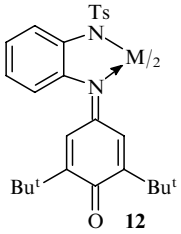


10: M = Zn, Cd.

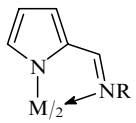


11: M = Co, Ni; L = H_2O.

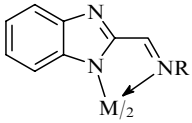
The quinone-imine groups can be involved in chelates **12** containing a five-membered coordination unit.[44–48]



12

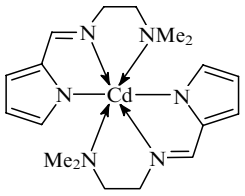


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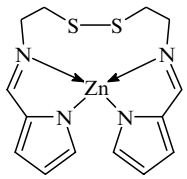


14

Complexes with Schiff bases of 2-formylpyrrole **13**[5, 12, 20, 21, 49–55] and 2-formylbenzimidazole **14**[43, 56–60] studied earlier belong to azomethine complexes containing the five-membered MN_4 coordination unit. It should be emphasised that chelates of type **13** were generated under the conditions of chemical[5, 12, 20, 21] and electrochemical[49–55] syntheses, whereas chelates **14** were prepared only by the electrochemical method[56–60] because of the rather low acidity of the N–H bond in benzimidazole. This approach was also employed in the synthesis of chelate compounds **15** and **16** with the use of pyrrole-based aldimine ligands possessing nitrogen-[55] or sulfur-containing[61] fragments. Tropone imine complexes also contain the MN_4 coordination unit with the azomethine bond.[20]



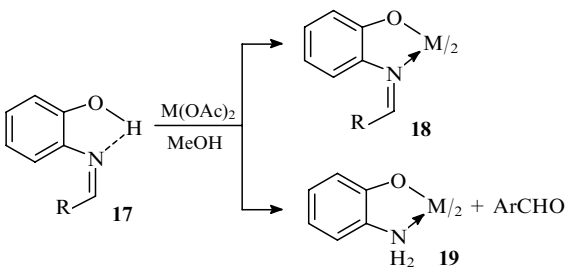
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16

b. Chelates with N,O-donor atoms

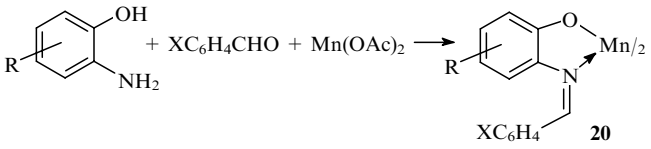
Azomethines **17**, which were derived from *o*-aminophenol and contain an intramolecular hydrogen bond, serve as bidentate ligands and form bicyclic chelate compounds **18** with the five-membered MN_2O_2 coordination unit.[20, 22, 25, 62, 63]



R = Ar, HetAr; M = Cu, Mn, Co, Ni, Zn, Cd *etc*.

These reactions, particularly those used for the preparation of copper(II) chelates[62] (**18**, M = Cu), are accompanied by partial hydrolysis of the exocyclic azomethine bond resulting in contamination of chelate compounds **18** with *o*-aminophenol complex **19**. Because of this, copper chelate **18** (R = Ph, M = Cu) was prepared in the presence of an excess of benzaldehyde.[62]

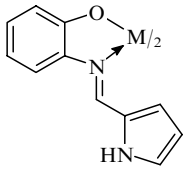
The template synthesis of manganese complexes **20**[64] is of interest.



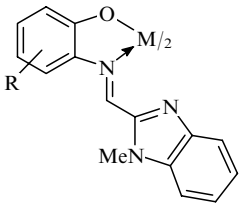
R = H, 4-NO_2; X = H, 2-NO_2, 3-NO_2, 2-MeO, 4-MeO, 4-Me_2N.

The reactions of compounds **17** with acetates of other metals (Co, Ni, Zn, Cd) in methanol afforded virtually pure complexes with composition ML_2.[20, 22, 24, 65, 66] The exceptions are the palladium chelates PdL_2, which were prepared[66] from azomethines **17** [R = $C_6H_4R^1$; R^1 = H, 4-Cl, 4-NMe_2, 2,4-$(OMe)_2$] in an Me_2CO – MeOH mixture.

Of complexes **18** with varying substituents R, noteworthy are chelate compounds containing heterocyclic fragments, for example **21**[67, 68] and **22**[69] prepared by chemical and electrochemical methods.

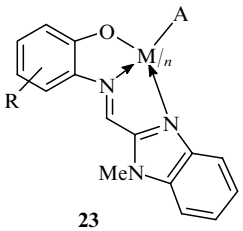


21: M = 3*d* metals.



22: R = H, 5,6-C_4H_4-cyclo; M = Co, Mn, Ni, Zn, Cd.

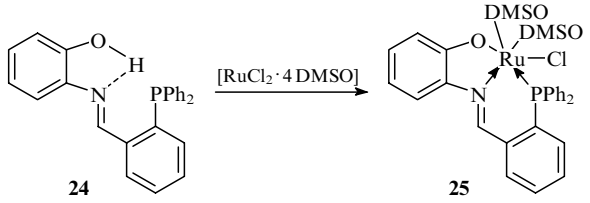
It was proposed (although this assumption was not supported by X-ray diffraction data) that complexes analogous to compounds **23**, which are generated from *o*-aminophenol and heteroaromatic aldehydes, can contain tridentate ligands.[70]



23

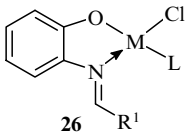
R = H, C_4H_4-cyclo; *n* = 1, 2; A is absent or is NO_3, OCOMe; M = Ni, Pb, Th, UO_2.

Compounds **17** whose aldehyde fragments possess substituents capable of being involved in additional coordination can serve as tridentate ligands as well.[71] Thus, the P,N,O-tridentate mode of coordination of the ligand was found in chelate **25**, which was prepared from compound **24** and the [$RuCl_2 \cdot 4$ DMSO] complex by the ligand-exchange reaction[24] in THF and structurally characterised.

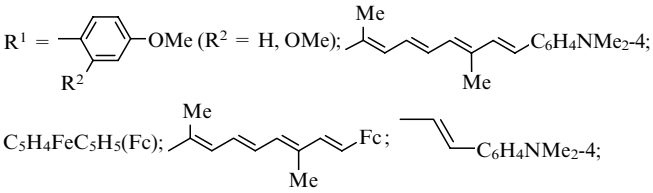


24 **25**

In most cases, bidentate ligands **17** are involved in tetracoordinated complexes with composition ML_2. However, monocyclic chelates **26** of the L^1L^2MCl type, which are composed of one monoanion of Schiff bases of *o*-aminophenol, were also synthesised[72] by the reactions of the corresponding ligands with the $[(R_3P)(Cl)M(\mu\text{-}Cl)]_2$ (M = Pd, Pt), $[(\eta^5\text{-}Cp)(Cl)Ir(\mu\text{-}Cl)]_2$ and $[(4\text{-}Pr^iC_6H_4Me\text{-}1)(Cl)Ru(\mu\text{-}Cl)]_2$ complexes in methanol.

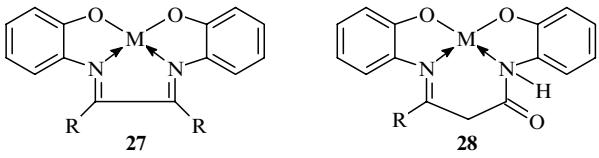


26



M = Pd, Pt; L = PR^3_3; R^3 = Et, Bu^n, Ph;
M = Ir; L = C_5H_4(Cp); M = Ru, L = 4-$Pr^iC_6H_4$Me-1.

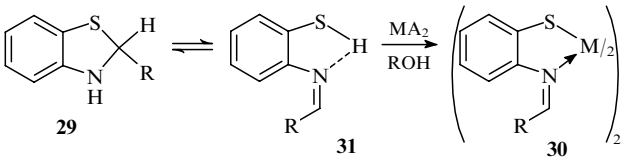
Recently,[73] complexes **27** and **28** containing three metallocycles each were prepared by the template synthesis.



27 **28**

c. Chelate compounds with N,S(Se)-donor atoms

The synthesis of chelates containing the five-membered MN_2S_2 coordination unit involves the well-studied reactions of Schiff bases of *o*-mercaptoaniline with metal salts.[20, 22, 24, 39, 74, 75]

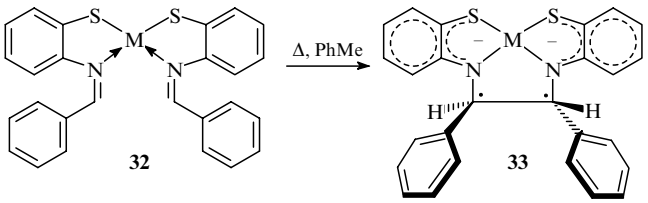


29 **31** **30**

R = Ar, HetAr, Fc; M = Co, Ni, Pt, Pd, Zn; A is an anion.

The reactions of Schiff bases of *o*-mercaptoaniline, which occur predominantly in the closed form of 2,3-dihydrobenzothiazole **29**,[76, 77] with metal salts in alcohols (methanol, ethanol) afforded chelate compounds **30** containing the open deprotonated form of *o*-mercaptophenylazomethine **31** as the ligand system. In these chelates, aromatic[78–87] or heterocyclic[39, 69, 75, 88–90] as well as organometallic systems (for example, ferrocene[74]) are most often involved as the substituent R.

Chelate compounds **30** exhibit a specific type of valence tautomerism.[83, 84] For example, heating of complexes **32** in toluene over a short period of time gave rise to cobalt and nickel 2,2′-bis(1,2-diphenylethylenediimine)benzothiolates **33** through the formation of the C – C bond.

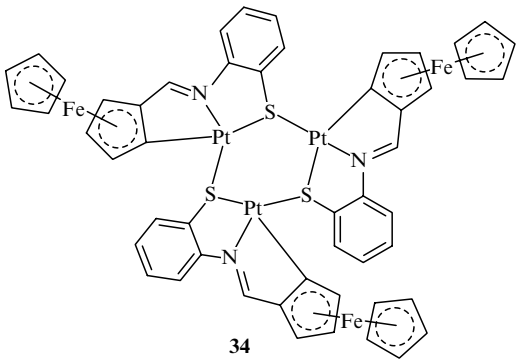


32 **33**

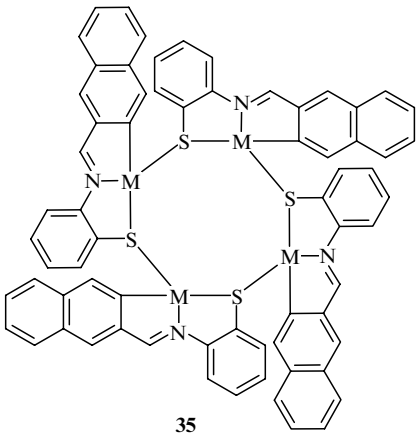
M = Co, Ni.

It should be noted that this transformation was not observed for chelates **30** containing the 3,5-di-*tert*-butyl-4-hydroxyphenyl fragments as the substituents R.[86]

In some cases, the reactions afforded tri- (**34**)[85] and tetrameric (**35**)[81, 82] structures along with complexes **30**.

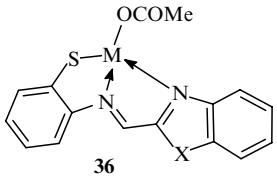


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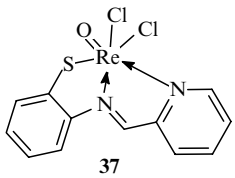


35

Most of chelates of tridentate azomethine ligands characterised by the N,S-ligand environment are azomethine complexes prepared from heterocyclic aldehydes.[39, 69, 75, 89–90] Compounds **36** (X = NMe,[39] S [69]) serve as examples of this type of chelate compounds.



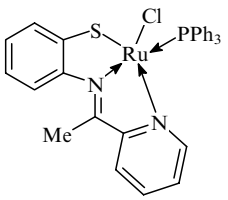
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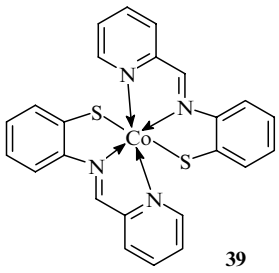
37

M = Pd, Zn, Cd; X = NMe, S.

2-(2-Pyridyl)-2,3-dihydrobenzothiazole [89] and 2-methyl-2-(2-pyridyl)-2,3-dihydrobenzothiazole [75] act as donor ligands of the N_2S type to give complexes **37** and **38**, respectively. 2-(2-Pyridyl)-2,3-dihydrobenzothiazole also serves as the tridentate ligand in tetracyclic chelates **39**.[90]

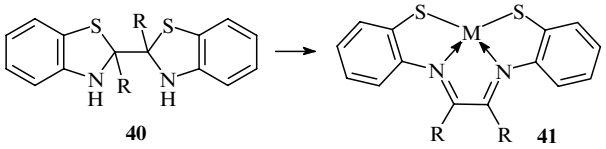


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39

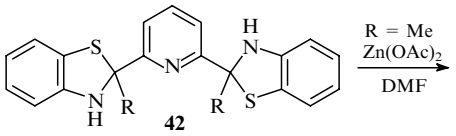
The complex formation involving bis-2,3-dihydrobenzothiazoles **40** gives rise to tricyclic chelates **41** with composition ML, in which mercaptoazomethines act as tetradentate ligands.[91]



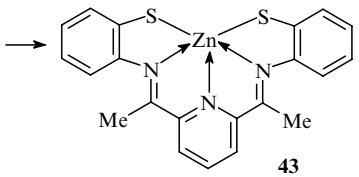
40 **41**

R = H, Me, Ph; M = Cd, Zn, Hg.

The use of 2,6-dicarbonyl derivatives of pyridine **42a**,**b** (LH_2 ligands) as the aldehyde (ketone) fragment made it possible to prepare chelates with pentadentate ligands.[88, 92, 93] The structure of complex **43** was established by X-ray diffraction analysis.[92]

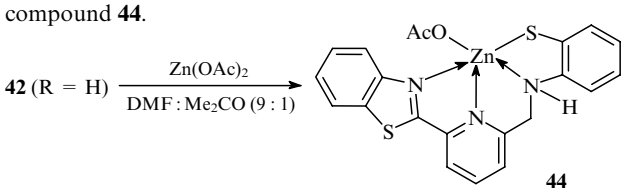


42



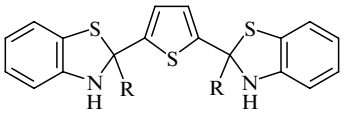
43

If the nature of the ligand system in compounds **42** (R = H) and the reaction conditions are changed,[94] the complex formation is accompanied by a redox process with the resulting changes in the type and denticity of ligand system **42** giving rise to chelate compound **44**.

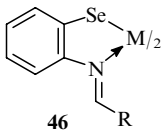


44

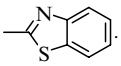
Apparently, chelate compounds prepared on the basis of 2,5-bis(2,3-dihydrobenzothiazol-2-yl)thiophene derivatives **45** [95] can also be assigned to five-membered azomethine chelates containing N,S-donor centres.



45



46

M = Zn, Cd; R = Ph, .

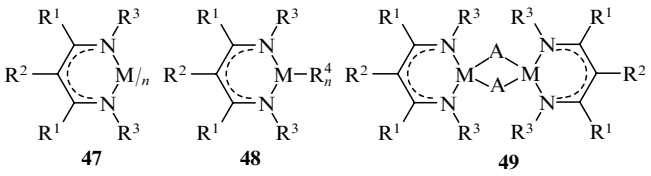
There are only a few complexes **46**, which belong to azomethine chelates containing five-membered metallocycles and the MN_2Se_2 coordination unit. These complexes were prepared by template synthesis from metal *o*-aminobenzeneselenolates and the corresponding aldehydes.[96]

Similar reactions involving the *o*-aminobenzenetellurol chelate compound [97] afforded metal-free ditelluride rather than a tellurium-containing chelate analogous to complex **46**.[98]

2. Chelate compounds with six-membered coordination units

a. Complexes with N,N-donor atoms

Not only numerous classical bis-β-aminovinyliminates **47** [4, 12, 17, 20, 22, 24, 99–104] but also monoanionic complexes **48** [101, 105–116] and binuclear structures **49** [108, 109, 112, 114, 117] are examples of six-membered metal chelates prepared on the basis of bidentate azomethine N,N-donors **1** (X = NR^5).



47 **48** **49**

A = Hal, OAc.

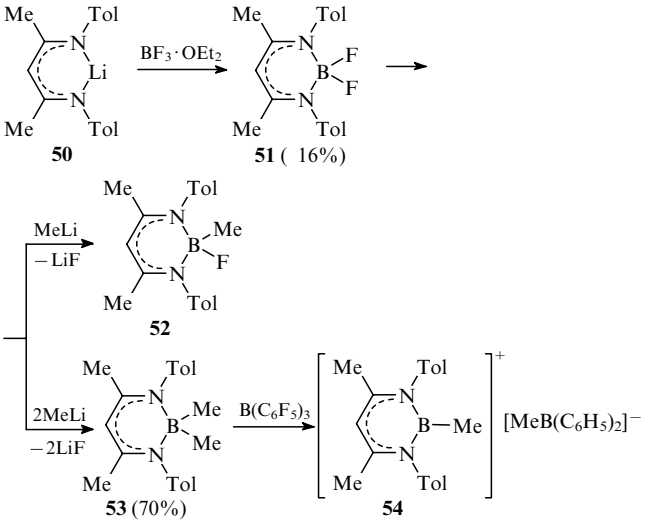
Most of chelate compounds **47** ($n = 2$) typical of β-aminovinylimines were obtained by the reactions of ligands **1** (X = NR^5) with metal salts in alcohols. In this connection, of particular interest is the synthesis of the first structurally characterised tris-diiminate complex **47** ($R^1 = R^3 = Me$, $R^2 = H$, M = Al, $n = 3$), which was prepared by the ligand-exchange reaction of 2-methylamino-4-methyliminopent-2-ene with $Me_3N \cdot AlH_3$ in toluene.[103]

First publications on complexes **48** appeared in the mid-1990's. These studies were devoted primarily to the directed synthesis of chelates, in which the metal centre is shielded by bulky substituents, and were aimed at constructing new catalysts of polymerisation and other processes.

β-Aminovinylimines **1** bearing bulky substituents (predominantly, C_6H_3-2,6-Pr^i_2) at the nitrogen atom were used for the preparation of this type of chelate compounds with lithium,[113] barium,[118] magnesium,[119–121] boron,[107] aluminium,[110,122–124] gallium and indium,[110] scandium,[125,126] samarium and gadolinium,[127,128] ytterbium,[129] tin,[130] titanium,[101,105,131] zirconium,[101,132,133] vanadium,[105] chromium,[105,134] copper,[106,109,111,114,115–117,135] zinc,[108,112,114] platinum[116] and rhodium.[136]

Chelates **48** were synthesised either from lithium complexes **50** (predominantly) or by the reactions of β-aminovinylimines with different complex-forming agents.

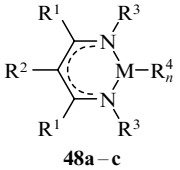
Thus, a series of boron-containing complexes of β-diketimines **51**–**54** were prepared.[107]



50 **51** (16%) **52** **53** (70%) **54**

Tol = 4-MeC_6H_4.

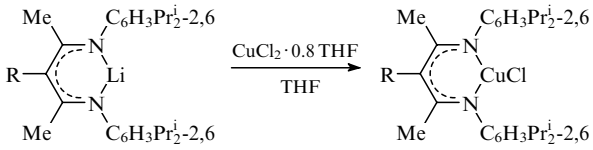
Chelate **51**, which was used as the starting compound in the synthesis of complexes **52**–**54**, was prepared by the reaction of $BF_3 \cdot OEt_2$ with lithium derivative **50** and isolated from a solution in toluene. β-Aminovinylimines were used for the synthesis of complexes of aluminium, gallium and indium **48a**,[110] titanium and zirconium **48b**[101] and chromium **48c**.[134]



48a–c

48a: R^1 = H, Alk, Ar; R^2 = H; R^3 = 2,6-$Pr^i_2C_6H_3$; R^4 = Me, Cl, I; M = Al, Ga, In; $n = 2$
48b: R^1 = Me; R^2 = H; R^3 = 4-MeC_6H_4; R^4 = Cl, NMe_2; M = Ti, Zn; $n = 3$
48c: R^1 = Me; R^2 = H, R^3 = 2,6-$Pr^i_2C_6H_3$; R^4 = $Cl(O_2CMe) \cdot THF$; M = Cr; $n = 1$

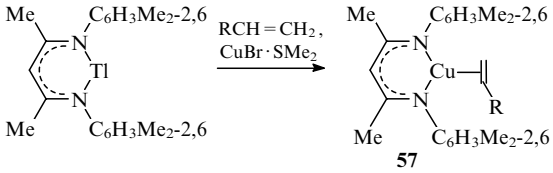
The metal-exchange method was also used for the preparation of copper complexes with β-aminovinylimines.[109,111,115] Noteworthy are the syntheses using lithium salts **55a**,**b** and the $CuCl_2 \cdot 0.8\,THF$ complex,[111] *i.e.*, procedures combining the metal- and ligand-exchange reactions.[24] These reactions afforded mononuclear chelate compounds **56a**,**b** containing the labile chlorine atom, which can be replaced by the OAr or CNAr groups.[111]



55a,**b** **56a**,**b**

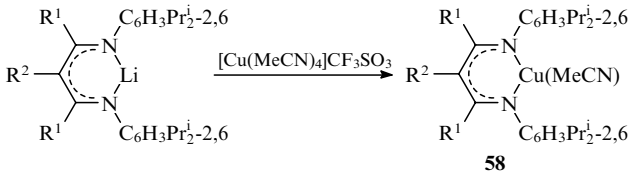
R = H (**a**), Me (**b**).

Monovalent copper chelates **57** and **58** were synthesised analogously.[109,115]



57

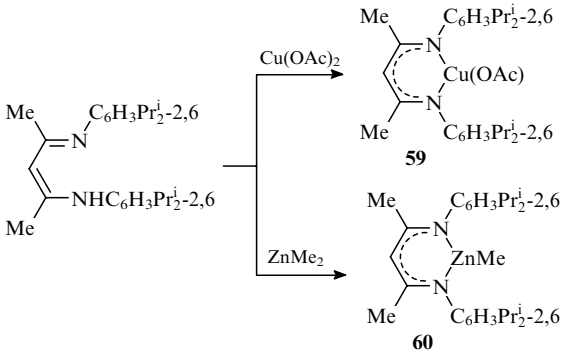
R = H, Ph.



58

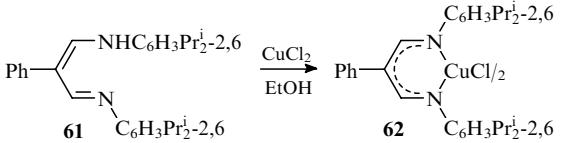
R^1 = H, Me, Bu^t; R^2 = H, Ph.

Copper chelate **59**[114] and zinc chelate **60**[112] were synthesised from the corresponding ligands and $Cu(OAc)_2$ or $ZnMe_2$, respectively. The structures of virtually all chelate compounds described above were established by X-ray diffraction analysis.



59 **60**

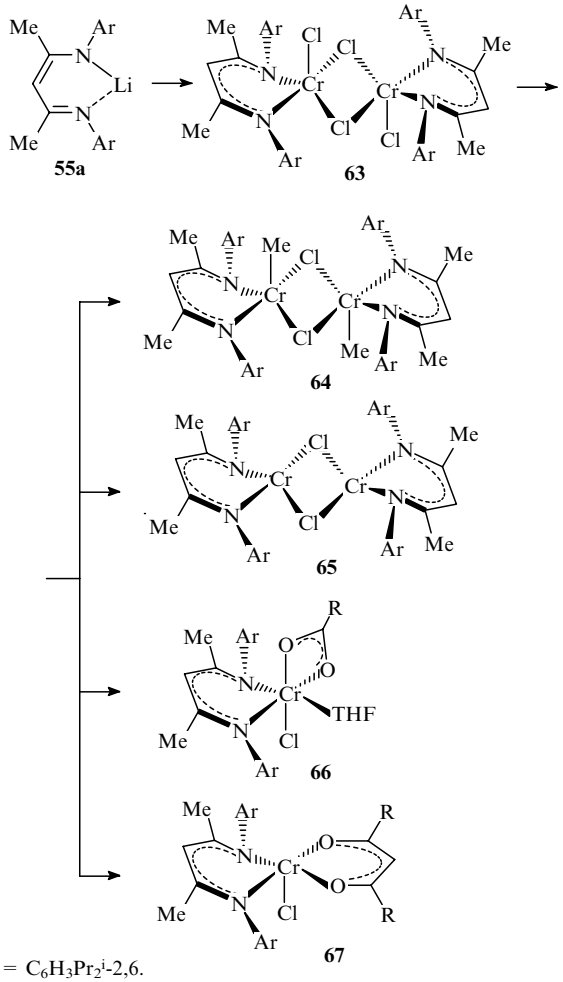
Binuclear complexes **49** were prepared primarily according to analogous methods, *viz.*, by the metal- or ligand-exchange reactions or with the use of a combined procedure. Thus, the reaction of aminovinylimine **61** with copper(II) chloride in alcohol gave rise to copper complex **62**, which was isolated and structurally characterised.[117]



61 **62**

Lithium complex **55a** was used for the preparation of bridged binuclear zinc chelates **49** (M = Zn; A = μ-Cl, μ-Br, μ-OAc).[108]

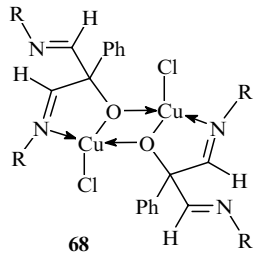
Binuclear complex **63**, which served as the starting compound for the preparation of chromium-containing chelates **64**–**67**, was prepared by the ligand-exchange reaction of lithium salt **55a** with the $CrCl_3 \cdot (THF)_3$ complex in THF.[134]



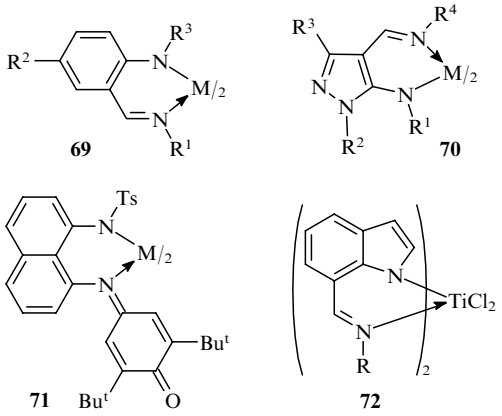
Ar = $C_6H_3Pr^i_2$-2,6.

In some cases, binuclear complexes **49** were synthesised with the employment of specific techniques. For example, structures **49** (R^1 = Me, R^2 = H, R^3 = Ar, M = Zn; A = H, F, OAc, OH, OMe) were prepared from mononuclear zinc complexes **48** [R^1 = Me, R^2 = H, R^3 = Ar, M = Zn; R^4 = Me, Et, $N(SiMe_3)_2$] and reagents, which were used for the formation of bridging fragments.[108, 112, 137, 138]

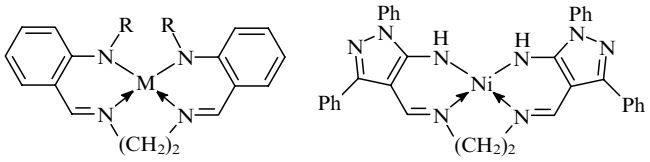
Recently,[139] it has been demonstrated that the reactions of β-aminovinylimines with $CuCl_2$ in alcohols can give rise to dimeric complexes **68** in which the aminoimine ligand is monodentately coordinated to the metal centre.



Chelates **69**,[17, 20, 22, 24, 39, 99, 140–145] **70**,[146] **71**[45, 147] and **72**[148] take an important place among chelate compounds containing the six-membered MN_4 coordination unit, which were prepared on the basis of N,N-donor bidentate azomethine ligand systems.



These complexes were synthesised predominantly by the direct reactions of the corresponding ligands with salts (predominantly, acetates) of 3*d* metals. In some cases, chelates **69** were prepared by template[140–142] or electrochemical[39, 143, 144] synthesis methods. It should be noted that azomethines based on N-unsubstituted 2-aminobenzaldehyde and aliphatic (aromatic) monoamines are unstable. Because of this, most of the syntheses were carried out with the use of 2-monotosylaminobenzaldehyde imines as precursors.[140–145] Complexes **72** (R = Ph, $C_6H_3F_2$-2,6, $C_6H_2F_3$-2,4,6) were prepared by the reactions of the lithium salt of 7-(*N*-aryliminomethyl)indole with titanium tetrachloride.[148] The Schiff base derived from 2-aminobenzaldehyde and ethylenediamine is quite stable. Hence, this Schiff base and sources of complex-forming metal were used for the preparation of chelate compounds **73a**[149] with tetradentate ligands.

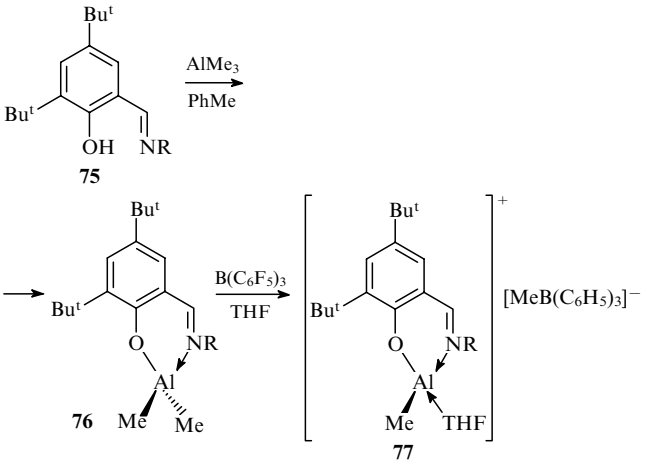


R = H, Me; M = Ni (**a**), Cu (**b**), FeCl(Br) (**c**).

Tricyclic chelates **73a**[149] were prepared by the reactions of the corresponding ligands with nickel acetate in methanol. Complexes **73b** were synthesised by the reaction of a suspension of $Cu(OMe)_2$ in methanol with the ligand. Chelates **73c** were prepared by the reactions of $FeCl_3 \cdot 6H_2O$ or $FeBr_3$ with the corresponding ligands in acetone.[150] The synthesis of nickel complex **74** was described in the study.[151]

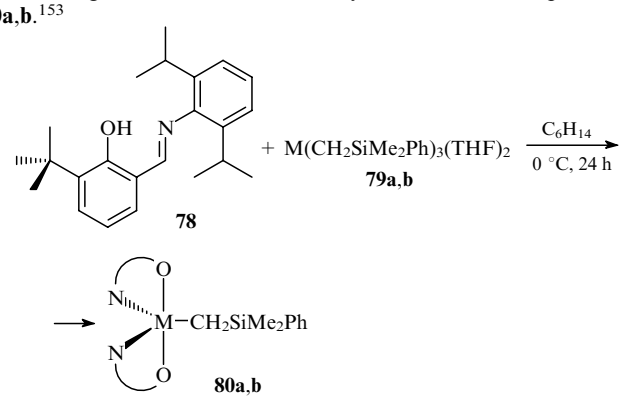
b. Chelate compounds with N,O-donor atoms

Metal chelates of bidentate hydroxyazomethines **1** (X = O) belong to the best-studied class of chelate compounds containing the six-membered MN_2O_2 coordination unit.[2, 4, 7, 9–16, 18–25] These compounds still attract interest today. Considerable attention is given to the synthesis and properties of chelate compounds with poorly studied metals, *viz*., aluminium,[152] scandium and yttrium,[153] titanium,[154–156] rhenium[157] and molybdenum.[158] Aluminium(III) chelate compounds were synthesised from azomethine **75** and Me_3Al. These reactions can afford not only neutral chelates **76** but also cationic complexes **77**. The structure of coordination compound **77** was established by X-ray diffraction analysis.[152]



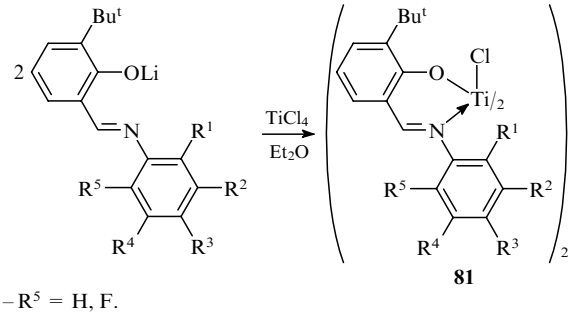
R = $C_6H_3Me_2$-2,6, $C_6H_3Pr^i_2$-2,6, $C_6H_3(CF_3)_2$-3,5 *etc.*

The reactions of Schiff bases **78** with organometallic derivatives **79a**,**b** gave rise to scandium and yttrium chelate compounds **80a**,**b**.[153]



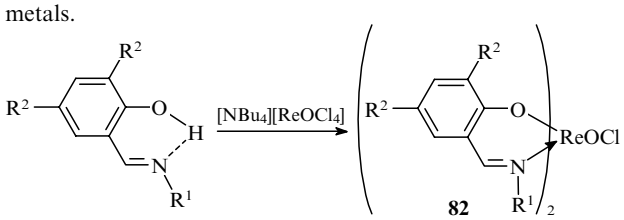
M = Sc (**a**), Y (**b**).

Titanium chelate complexes **81** were synthesised by the metal-exchange method from lithium salts of the ligands and titanium tetrachloride.[154–156]

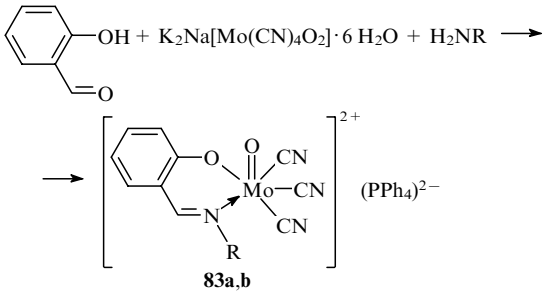


R^1–R^5 = H, F.

Chelates of Re(V) **82**[157] and Mo(VI) **83a**,**b**[158] were prepared with the employment of unusual sources of complex-forming metals.



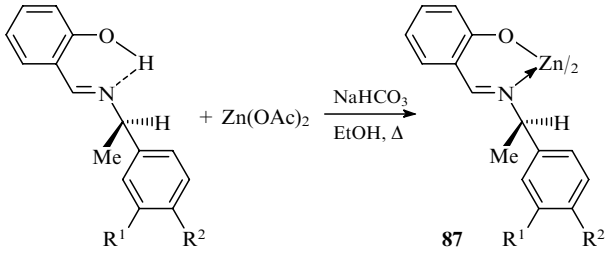
R^1 = Ph, cyclo-C_6H_{11}; R^2 = H, Cl.



R = Me (**a**), $(CH_2)_nNH_2$; *n* = 2, 3 (**b**).

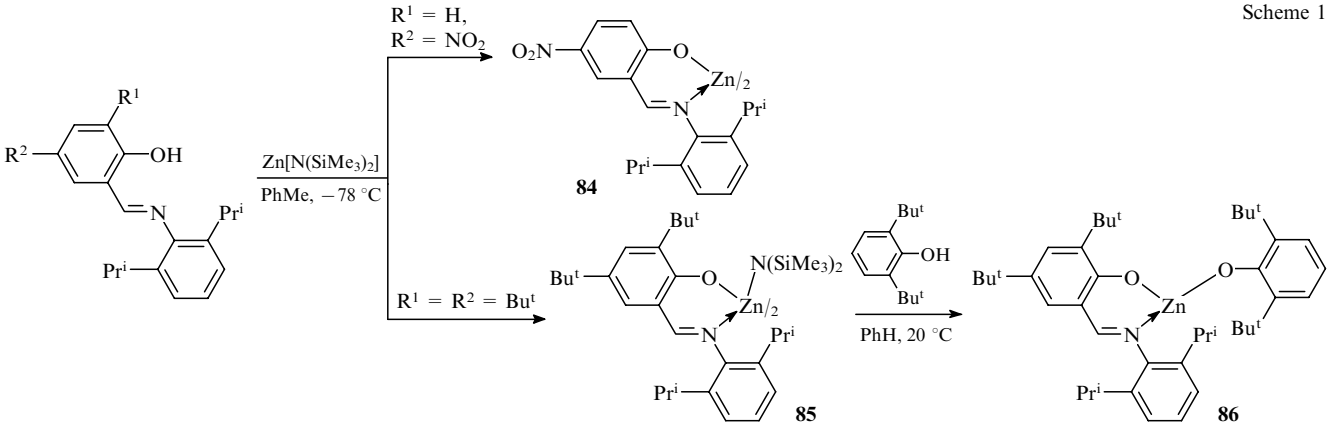
An important line of investigation in the chemistry of complexes with hydroxyazomethine ligands deals with the synthesis and studies of chelate compounds **2** (X = O) containing various substituents R^1–R^3, including fragments annelated to the metallocycle. As an example we refer to the synthesis of zinc complexes **84**–**86** (Scheme 1).[159]

The reactions of azomethines, which were prepared from salicylaldehyde and chiral α-phenylethylamines, with zinc acetate afforded optically active zinc complexes **87**.[160]

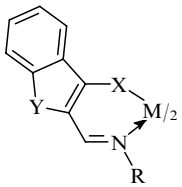


Metal chelates in which heterocyclic fragments are annelated to metallocycles were examined in the studies.[20,22] For example, benzofuran derivatives of chelate compounds **88** were prepared.[161,162] Chelates **89** and **90** were synthesised on the basis of β-aminovinyl ketones[163–168] or *o*-hydroxybenzaldimines[169,170] and the nitroxyl imidazoline radicals and their magnetic properties were studied. Complexes **89** and **90** were prepared

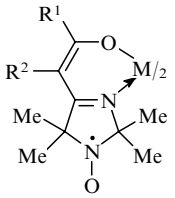
Scheme 1



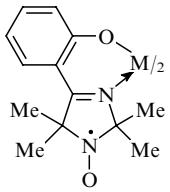
predominantly from the corresponding ligands and metal acetates in aqueous ethanol.



88: X, Y = O; R = Alk, Ar; M = Co, Ni, Cu



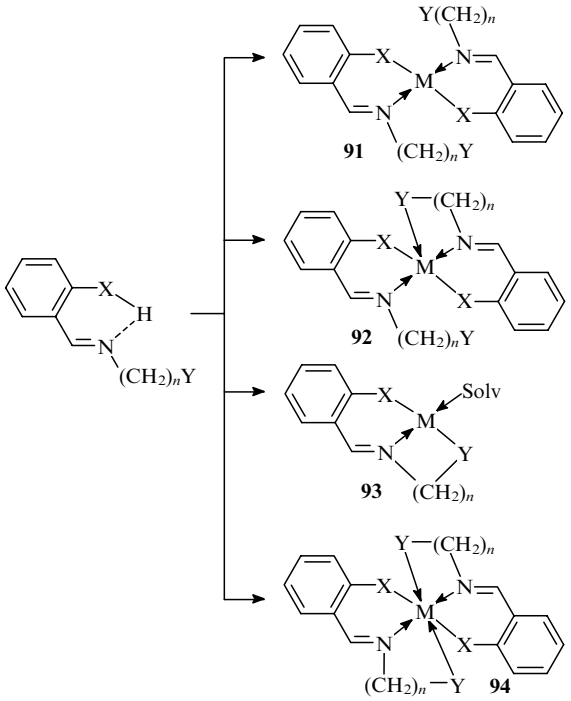
89: R^1 = H, Me; R^2 = Alk, CF_3, Ph, CO_2Et, $CONH_2$; M = Co, Ni, Cu, Zn, Hg, Cd



90: M = Co, Ni

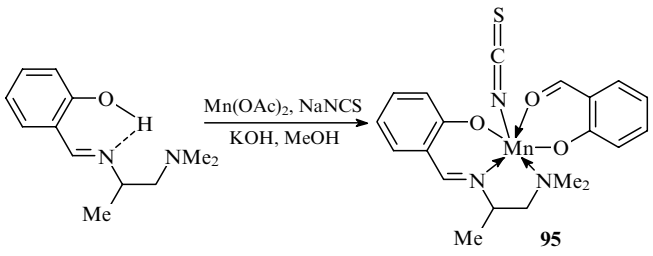
In some cases, *o*-hydroxyazomethine chelates were synthesised by the electrochemical method.[24, 43, 60, 171, 172]

An increase in denticity of the hydroxyazomethine ligands can be achieved by the introduction of the substituents R containing centres which can be involved in additional coordination, in particular, the alkylamino, hydroxy, alkoxy or alkylthioalkyl fragments as well as their aryl analogues. Such ligand systems are involved in complexes **91**–**94**.[12, 24]

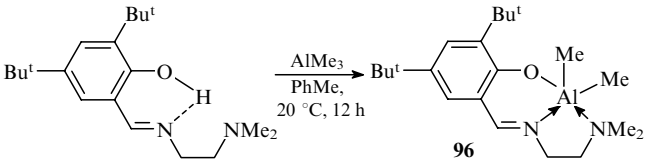


X = O; Y = NR_2, OR, SR; R = H, Alk, Ar; *n* = 2–4.

Salicylaldehyde-based azomethine complexes have been studied for many years.[12, 173] In these complexes, additional coordination can occur through the dimethylaminoalkyl substituents. For example, a ligand of this type is present in the structurally characterised manganese chelate **95**.[174]

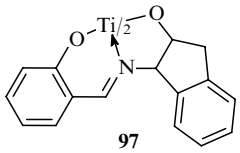


This type of metal binding was observed in complex **96**.[175]

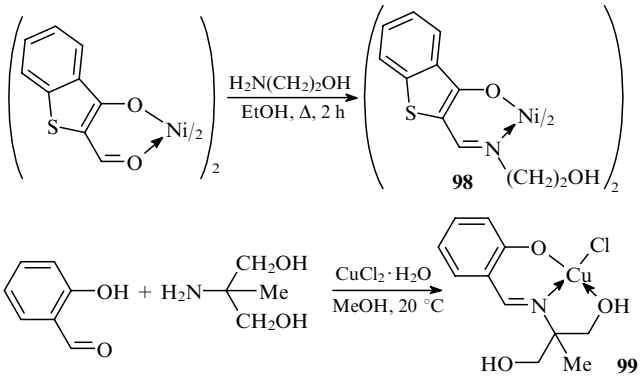


By contrast, X-ray diffraction analysis demonstrated that the nitrogen atom of the aminoalkyl substituent is not involved in additional coordination in molybdenum complexes **83b** synthesised by the template method.[158]

Among chelate compounds **91**–**94**, complexes of salicylideneamino alcohols have received the most study. Early investigations devoted to their synthesis, structures and properties have been surveyed in reviews.[176–178] Recently, chelate compounds of titanium(IV),[179] vanadium(V),[180, 181] dioxomolybdenum(VI),[180, 182, 183] cobalt(III),[184] nickel(II)[185–187] and copper(II)[186] were prepared and characterised. Titanium complexes of type **93** containing the deprotonated OH groups were synthesised from the ligands and $Ti(OPr^i)_4$.[179] Complexes **93** with vanadium[180, 181] and molybdenyl[182, 183] were prepared by the ligand-exchange reactions with the use of acetylacetonates of these metals. As an example we refer to compound **97**.



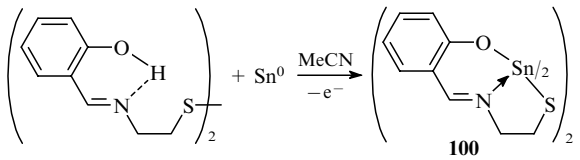
The structures in which the OH groups are retained are typical of nickel (**98**)[186] and copper (**99**)[187] complexes synthesised by the template method.



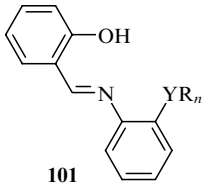
In the fluorine-containing barium β-ketoiminate complexes $Ba[CF_3COCH=C(NR)CF_3]_2$ [R = $CH_2(CH_2CH_2O)_2$, $(CH_2)_2.(CH_2CH_2O)_2$ or $CH_2CH(CH_2CH_2O)_3$], which were prepared from the corresponding ligands and BaH_2 in a mixture of heptane and THF,[188] the metal atom is coordinated by the oxygen atoms of the alkoxy groups.

Most of the complexes prepared on the basis of mercaptoalkylimines are bi-[189, 190] or trinuclear.[190] However, Zelentsov and Suvorova[191] expected that the mononuclear nickel complex could be isolated.

Iminothiol complexes **100** were prepared by the electrochemical cleavage of the disulfide bond.[192]



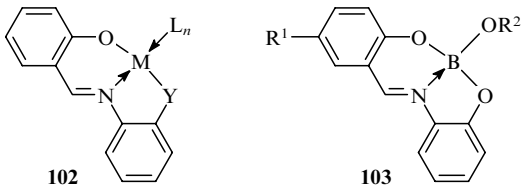
Salicylideneanilines **101** containing various proton- and electron-donating groups YR in the *ortho* position of the *N*-aryl fragment are very widespread azomethine tridentate ligands.



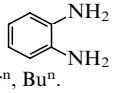
101

Y = NTs, P, O, S;
R = H, Alk, Ar.

Although most of the complexes prepared on the basis of ligands **101** have binuclear structures (see Section IV), mononuclear chelates (predominantly of type **102**) were prepared with the employment of some complex-forming agents. Thus, mononuclear complexes of boron,[193] aluminium,[175] tin,[194,195] rhenium,[196–198] molybdenum,[199] tungsten,[199] cobalt,[200–204] nickel,[201–208] copper[201,203,207,208] and cadmium[202,209] are known. A series of chelate compounds under consideration were synthesised by the direct reactions of compounds **101** with different sources of metals, *viz*., $AlMe_3$ (in PhMe), tin, nickel, zinc, copper and cadmium salts (in alcohols). Analogously, boron complexes **103** were synthesised by the reactions with $B(OR)_3$, where R^2 = Me, Et, Pr or Bu^n, in a mixture of toluene and THF.[193]

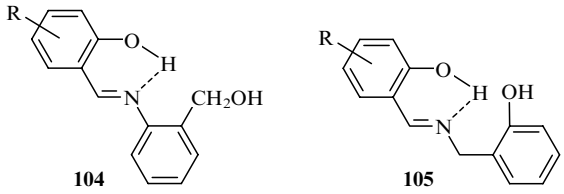


102 **103**

102: Y = NTs, P, O, S; L = Py, bipy,  *etc*..; *n* = 1, 2;

103: R^1 = H, Cl, Br; R^2 = Me, Et, Pr^n, Bu^n.

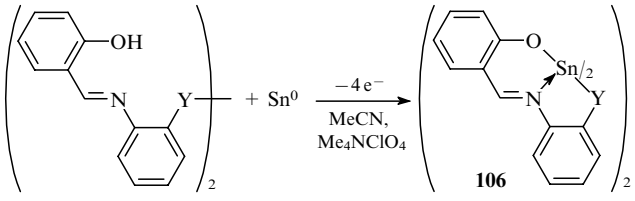
Oxorhenium chelates **102** (M = ReO) were prepared by the ligand-exchange reactions of $[ReOCl_3(PPh_3)_2]$, compounds **101** and ligands L. In particular, 2-(2-hydroxyphenyl)benzoxazole,[197,198] 2,3-dihydro-2-(2-hydroxyphenyl)benzothiazole[197] and triphenylphosphines containing the proton-donating group (OH or CO_2H) in the *ortho* position of one of the benzene rings[198] were used as the ligands L. Dioxomolybdenum and dioxotungsten complexes were prepared[199] from Schiff bases **104** and **105** and acetylacetonates of MoO_2 and WO_2, respectively (in MeOH).



104 **105**

R = H, 5,6-C_4H_4-cyclo.

The electrochemical method is widely employed for the preparation of chelate compounds **102**.[200–204,207] This method proved to be particularly useful in the case of chalcogen-containing tridentate azomethines. Thus, chelates **106** were synthesised from the corresponding disulfides and diselenides.[194,210]

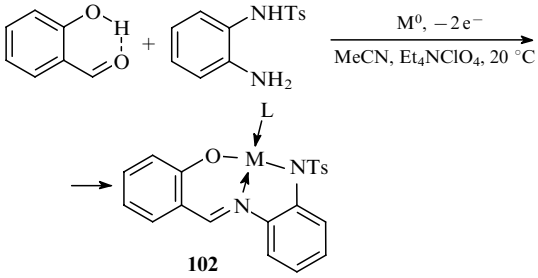


106

Y = S, Se.

An important advantage of this method is that it allows one to use various N-donor ligands,[200–208] including ambidentate 1-alkyl-2-aminobenzimidazoles.[201–204]

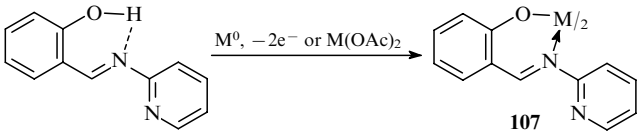
Chelates **102**, like other complexes, can be prepared by the template electrochemical method.[202]



102

M = Ni, Zn; L = H_2O, MeCN.

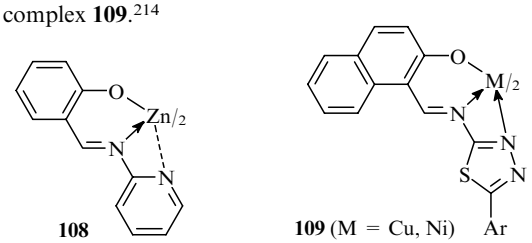
It should be noted that the donor centres Y in azomethines **101**, like those in other compounds containing N-donor fragments at the nitrogen atom,[24] are not necessarily involved in coordination to the metal atom. Thus, X-ray diffraction analysis demonstrated that the nitrogen atom of the pyridyl fragment in chelates **107**, which were obtained from the potentially tridentate pyridine-substituted azomethine by the reactions with metal salts or under the conditions of the electrochemical synthesis, is not bound to the metal atom.[211,212]



107

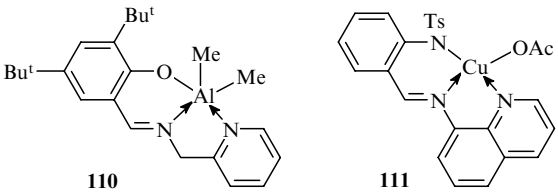
M = Cu, Ni.

By contrast, X-ray diffraction analysis revealed the $Zn-N_{py}$ interaction (the distance is ~ 2.8 Å) in the analogous zinc chelate **108**.[213] It was suggested that additional coordination also occurs in complex **109**.[214]



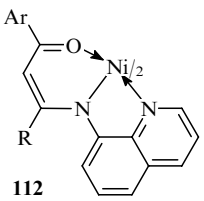
108 **109** (M = Cu, Ni)

The difference in the behaviour of the coordinatively active pyridyl substituent in complexes **107** and **108** was attributed to the instability of four-membered metallocycles.[215] Actually, stable bicyclic chelate compounds with tridentate ligands were obtained under conditions favourable for the formation of five-membered coordination units due to the insertion of the methylene group (complex **110**)[175] or the quinoline substituent (complexes **111**[216] and **112**[217]).

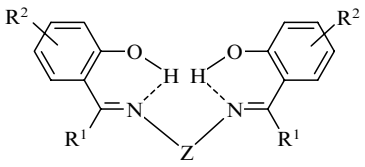


110 **111**

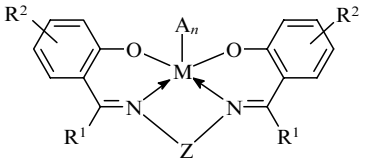
Bissalicylidene derivatives of aliphatic, aromatic and heterocyclic diamines **113**[2,9,12,20–22,24,218] serve as tetradentate N,O-donor azomethine ligands, which gave rise predominantly to tricyclic chelates **114**.



112



113



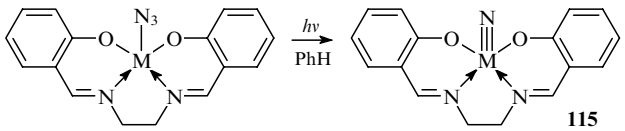
114

R^1 = H, Alk; R^2 = H, Alk, Ar, Hal, NO_2, OAlk; A = Alk, Hal; Z = $(CR^3R^4)_m$, Ar, HetAr; R^3, R^4 = H, Alk, Ar; n = 0, 1, 2.

In recent years, chelate compounds **114** with aluminium,[218–221] gallium,[218,222] indium,[218,222–225] tin,[226] titanium,[227] manganese,[228–237] vanadium,[238,239] chromium,[230,240] molybdenum,[241] cobalt,[236,241] iron,[231,236,241] nickel,[231,236,241,242] ruthenium,[243–245] palladium,[246] osmium,[247] copper[231,236,246,248–250] and zinc[251,252] have been prepared. Aluminium, gallium and indium complexes were synthesised by the reactions of ligands **113** with alkyl and other derivatives of these metals.[218]

Chelate compounds of Group III metals were prepared with the use of a mixture of acetonitrile and THF[221] or cyclohexane[222] as the solvent. Tin complexes of the general formula R_2SnL (R = Me, Bu^n, Ph) were synthesised in benzene with the addition of triethylamine under an atmosphere of nitrogen starting from the corresponding tin(IV) dichloride and tetradentate Schiff bases derived from salicylaldehyde, 3-methoxysalicylaldehyde, 4-benzoyl-3-methyl-1-phenylpyrazol-5-one, *o*-phenylenediamine and 1,3-propylenediamine.[226]

Complexes **114** (M = Mn: A is either absent[236] or is ClO_4,[228,233] Cl,[229,231,237] H_2O;[229] M = Cr: A = Cl[240]) were prepared from ligands **113** and the corresponding manganese or chromium salts in alcohol. Adducts of bis(salicylidene)ethylenediamine chelates of manganese and chromium with atomic nitrogen (**115**) were synthesised from azido complexes.[230,253]

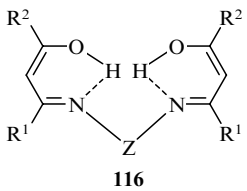


115

M = Mn, Cr.

Osmium nitride complexes **114** [M = Os: R^1 = H, R^2 = H, Cl, Me, OMe; A = N, MeOH; Z = $(CH_2)_2$] were prepared by the reactions of the corresponding ligand **113** with $[NBu_4][Os(N)Cl_4]$ in MeCN or MeOH.[247]

The direct reactions of the reagents were used in the synthesis of chelates **114** containing nickel (M = Ni; A is absent),[242] ruthenium (M = Ru; A = Cl),[244] copper [M = Cu; A is absent; R^2 = 5-(N=NPh),[249] OMe[250]] or zinc [M = Zn; A = THF, Py; R^2 = 3,5-Bu^t, 3-Bu^t-5-(4-Py); L = Py or absent].[251,252] Ruthenium chelate compounds were also prepared by ligand exchange in the $RuCl_2(PPh_3)_3$ complex.[245] The same synthetic approach

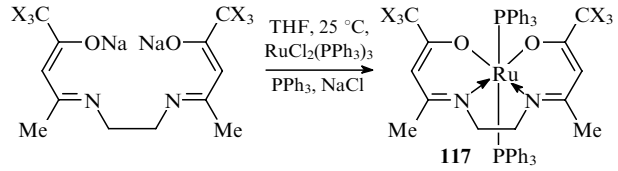


116

R^1 = Me, CF_3; R^2 = H, Me, CMe_3; Z = $(CH_2)_2$, $(CH_2)_3$, $MeCHCH_2$, $\overbrace{CH(CH_2)_4CH}$.

was applied in the preparation of chelates of Ru(II)[254] and Ru(III)[255] from bis-β-aminovinyl ketones **116**.

Ruthenium chelate compounds **117** were synthesised by combining the ligand- and metal-exchange reactions[254] with the use of the triphenylphosphine complex $RuCl_2(PPh_3)_3$ and its arsine analogues $RuY_2(AsPh_3)_3$ (Y = Cl, Br) as the source of metal.[255]

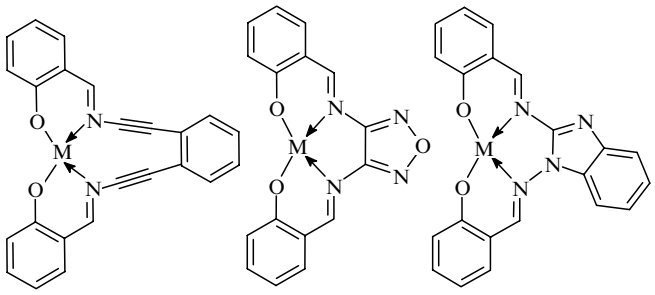


117

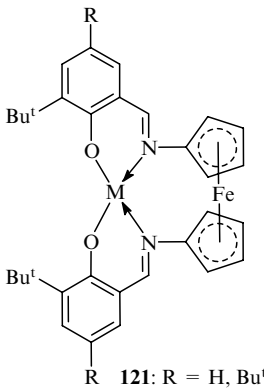
X = H, F.

Copper and nickel chelate compounds with ligand **116** [R^1 = H; R^2 = C_6H_4OMe-4; Z = $(CH_2)_3$, 1,2-C_6H_{10}-cyclo] were synthesised in methanol with the use of acetates of the corresponding metals.[256]

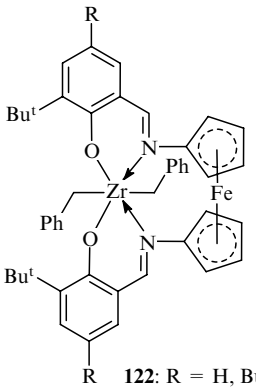
A series of studies were devoted to modifications of ligand systems of type **113** by varying the Z bridge (for example, complexes **118**,[257] **119**,[258] **120**,[259] **121**[260] and **122**[260]) and aldehyde fragment (complexes **123**[226] and **124**[261]).



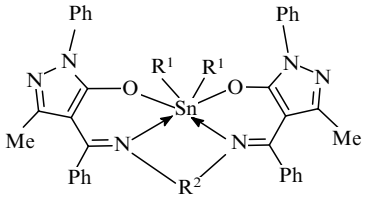
118: M = Ni, Cu. **119**: M = Ni, Cu, Co, Zn, Cd. **120**: M = Ni, Cu, Co, Zn, Cd.



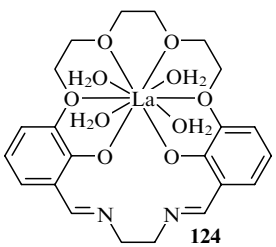
121: R = H, Bu^t.



122: R = H, Bu^t.

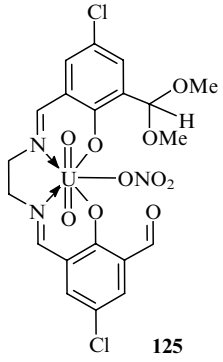


123: R^1 = Bu^n, Ph; R^2 = $(CH_2)_3$, C_6H_4-*o*.

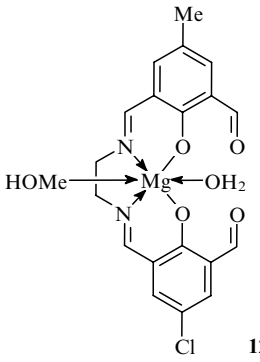


124

It was demonstrated[262–264] that potentially penta- or hexadentate N,O-donor azomethine systems containing the bis(salicylidene)ethylenediamine fragment, like the above-considered compounds **118**–**123**, form chelates with tetracoordinated ligands, such as complexes **125**[262] and **126**.[264] Chelates **117** and **120**–**125** were synthesised from the corresponding ligands and various metal sources. Complex **118** was prepared by the template method.



125



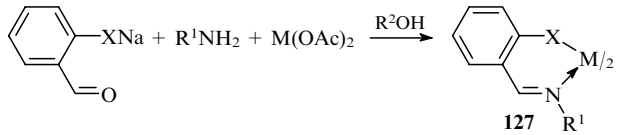
126

According to the X-ray diffraction data,[261] the ligand in complex **124** is coordinated through the crown fragment rather than through the azomethine group. It should be emphasised that ligands in complexes **124**–**126** can be considered as ambidentate ligand systems.[27] Taking into account that the lanthanum cation is the hardest acid of all the cations involved in these complexes,[24] the structure assigned to complex **124** seems to be justified.

c. Chelate compounds with N,S(Se)-donor atoms

In 1968, the template synthesis (method *a*) of metal chelates **127** of sulfur-containing bidentate Schiff bases generated from the sodium derivative of *o*-mercaptobenzaldehyde was carried out by two independent research groups.[265, 266] These compounds were the first representatives of azomethine chelates containing the MN_2S_2 coordination unit.

Method *a*

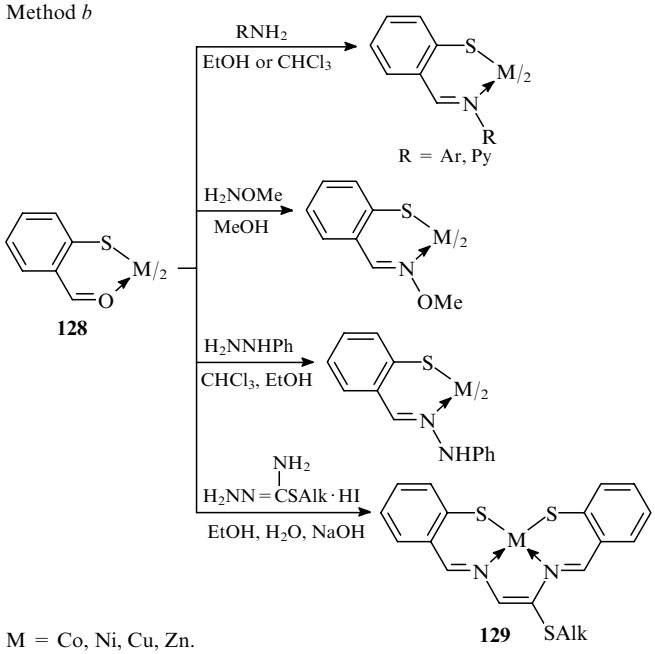


127

X = S, Se; R^1 = Alk, Ar, AlkO *etc.*; R^2 = Me, Bu^n;
M = Co, Ni, Cu, Zn *etc.*

Complexes **127** are also often prepared by the reactions of stable mercaptosalicylaldehyde chelates with amines and their derivatives (method *b*).[20, 24]

Method *b*



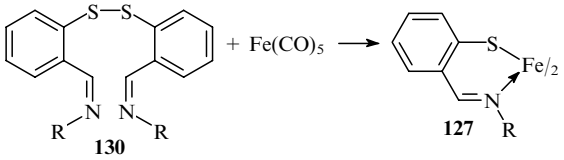
128 **129**

M = Co, Ni, Cu, Zn.

The use of chelates **128** enables one to construct coordination compounds **127** containing various substituents at the nitrogen atom, *viz.*, aryl,[267, 268] pyridyl,[269] methoxy [270] and phenylamino groups,[271] as well as alkylthiovinyl-bridged complexes **129**.[272]

The method *a* is more universal than the method *b* because it allows one to prepare not only chelate compounds **127** containing the MN_2S_2 coordination unit but also complexes of Schiff bases with the MN_2Se_2 chelate unit.[273]

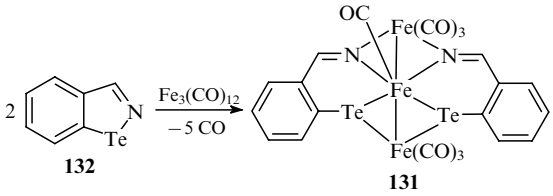
Chelates **127** can also be prepared starting from bisazomethine disulfide **130** and iron pentacarbonyl.[274]



130 **127**

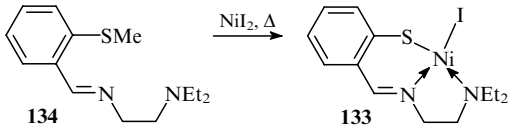
R = C_6H_4OMe-4.

Complex compounds **131** synthesised from benzotellurazole **132** can be considered as tellurium-containing analogues of chelates **127**.[275]



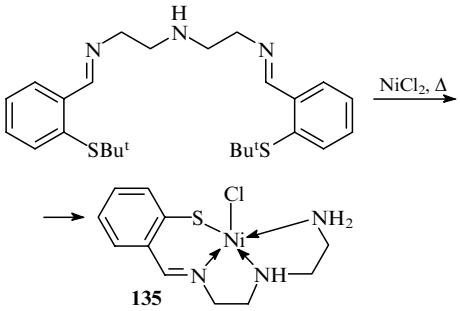
132 **131**

Tridentate sulfur-containing azomethine complexes **133** were first prepared according to an original procedure employing the reactions of nickel iodide with Schiff base **134**.[20]



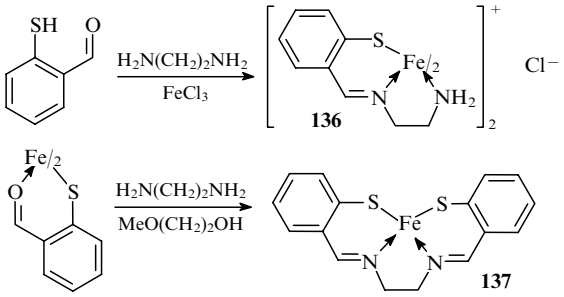
134 **133**

Tetracoordinated nickel complexes **135** were synthesised analogously.[276]

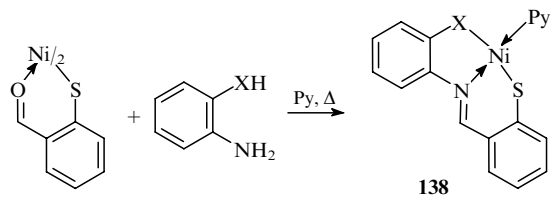


135

Complexes **136**,[277] **137** [274] and **138** [278] were constructed by the template method.



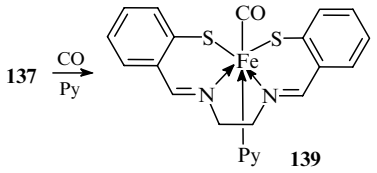
136 **137**



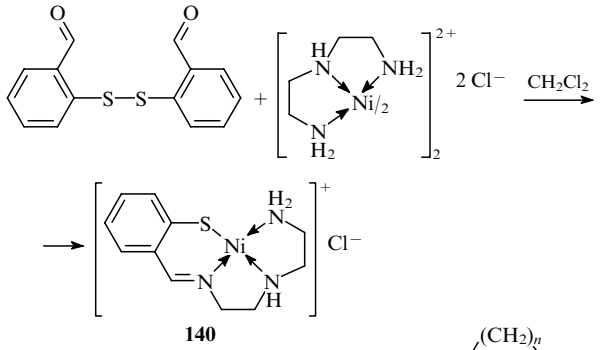
138

X = NTs, O, S.

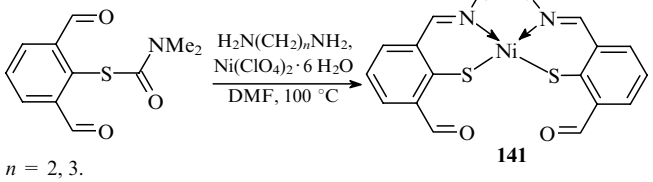
The template synthesis [279] was used for the preparation of chelates of tetradentate N,S-donor azomethine ligands **139**,[274] **140** [280] and **141**.[281, 282]



139



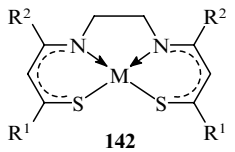
140



141

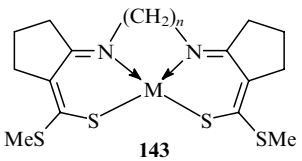
n = 2, 3.

The synthesis and structures of the structural analogues of these chelate compounds, *viz*., chelates **142** and **143**, were described in the studies cited in review [20] and monograph.[24]



142

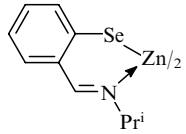
M = Co, Ni, Zn;
R^1 = Alk, Ar; R^2 = H, Alk.



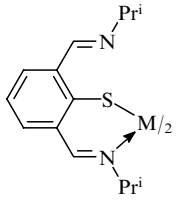
143

n = 2–4; M = Ni, Cu.

Let us emphasise that the rational design of chelate compounds of azomethine N,S(Se)-donors is of fundamental importance in the synthesis of complexes with selenium-containing ligands (for example, **144** [283]) or complexes **145** [20] containing alterdentate ligands, which are difficult to prepare.[284–286] The latter complexes play an important role in studies of the stereodynamics of tetracoordinated azomethine structures.[286]

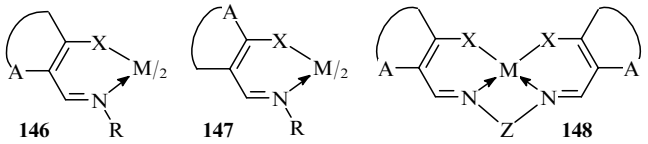


144



145: M = Zn, Cd.

Investigations of the synthesis, structures and properties of chelate compounds of heteroaromatic Schiff bases containing N,S(Se)-donor centres **146**–**150** were started in the late 1960s–early 1970s.[8, 161, 287–290]

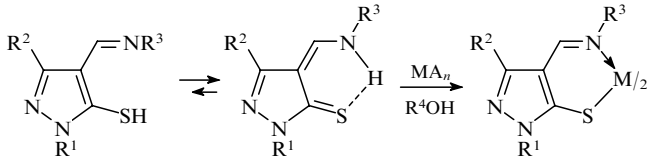


146 **147** **148**

A = O, S; X = S, Se; M = Co, Ni, Cu, Zn.

These complexes have still attracted attention in subsequent years.[20, 22, 24, 267, 269, 291–307]

Azomethines derived from thio-substituted heterocyclic aldehydes (for example, compound **149a**), which exist in the thioneamine form **149b**,[77] are more stable than their aromatic analogues. Because of this, chelate compounds **150** are synthesised primarily by direct reactions of the corresponding ligands with metal salts.[24]

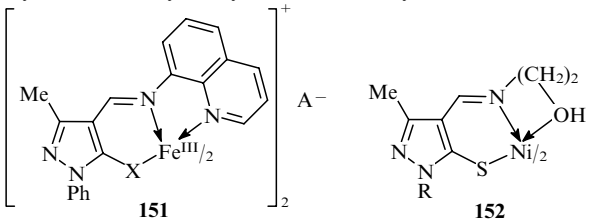


149a **149b** **150**

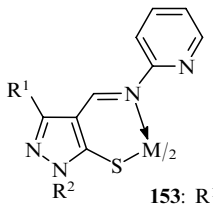
In some cases, chelates of type **88** (X = S, Y = O) were prepared by template synthesis.[161]

Of the chelate compounds under consideration, complexes **150** containing the pyrazole fragment annelated to the metallocycle deserve most attention.[20, 288–307] In these complexes, *d* metals (Co, Ni, Pd, Cu, Zn, Cd, Hg) act as complex-forming agents.[20] The synthesis of copper complexes of this type has presented substantial difficulties for many years. The first representative of these complexes (**150**, M = Cu; R^1 = Pr^i; R^2 = Me, R^3 = 2-Py) was prepared as late as 1992.[269] Later on, these chelates became readily available.[299–301, 303–307]

Some chelate compounds with tridentate ligands **149** [R^3 = $(CH_2)_2OH$,[185, 308] 2-pyridyl,[269, 294, 309] 8-quinolyl [298–300]] were synthesised. The structure of compound **151** was unambiguously established by X-ray diffraction analysis.[298]



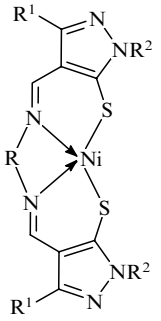
151 **152**



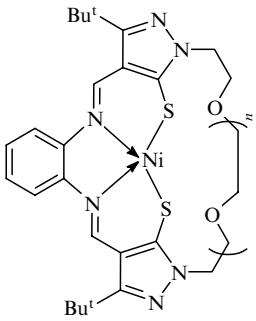
153: R^1, R^2 = Alk, Ar; M = Ni, Cu.

The coordination of the oxygen atom of the hydroxy group to the metal atom is observed in complex **152**.[185] By contrast, the potentially tridentate pyridine-containing ligand in compound **153** is in fact (according to the X-ray diffraction data) bidentate.[269, 292, 294]

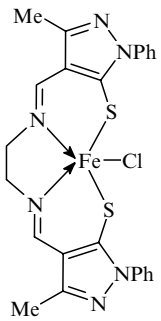
Nickel complexes of **154** [296] and **155** [291] and iron complex **156** [298] can be cited as examples of chelates with tetradentate N,S-donor ligands. These chelates were synthesised by the reactions of the corresponding ligands with metal salts [$Ni(OAc)_2$, MeOH; $FeCl_3$, EtOH].



154: R = Alk; R^1, R^2 = Me, Ph.

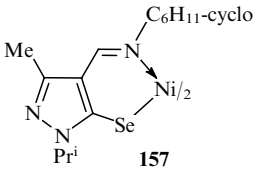


155: n = 1–5.

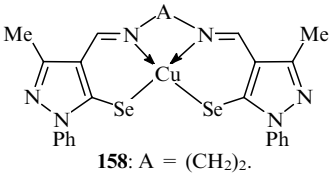


156

Examples of heteroaromatic azomethine chelates containing the six-membered MN_2Se_2 coordination unit are known, among which are complexes of nickel **157** [309] and copper **158**.[304]

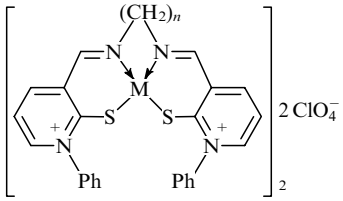


157

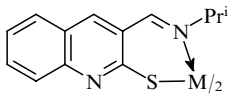


158: A = $(CH_2)_2$.

In conclusion, it should be noted that in searching for azomethine ligands on the basis of heterocyclic aldehydes, researchers were oriented primarily to five-membered heterocycles, *viz*., furan, thiophene, their benzo analogues and azole systems. However, chelate compounds, such as complexes **159** [310] and **160**,[311] can also be synthesised.



159: n = 2, 3.



160: M = Zn, Cd.

These complexes contain Schiff bases as chelating ligands, which were prepared from six-membered azine heteroaromatic aldehydes.

Chelates **160** are of importance in the investigation of the problem of the stereochemical non-rigidity of azomethine metal complexes.[285, 286]

3. Structures of azomethine metal chelates

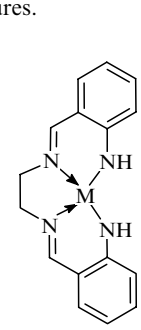
The structures of most of the above-considered chelates were unambiguously established by X-ray diffraction analysis. The detailed discussion of X-ray diffraction data is beyond the scope of the present review. Let us only briefly consider the structural aspects of the directed construction of azomethine metal chelates of a desired stereochemistry by varying the fine structure of ligand systems and electronic configuration of complex-forming metals.

Although data from X-ray diffraction analysis are available only for a limited number of five-membered metal chelates formed by bidentate Schiff bases, they provide evidence that chelate compounds containing the MN_4 or MN_2O_2 coordination units are tetrahedral.[20, 22, 24, 65] Metal chelates containing the MN_2S_2 coordination units (M = Ni, Pd) have planar structures with a *cis* arrangement of the ligands.[79–86, 312]

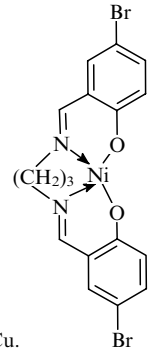
The structures of azomethine chelate complexes containing six-membered metallocycles and the same set of donor centres were studied in more detail.[2, 4, 12, 14, 20–22, 313–319] It was demonstrated that tetrahedral or distorted tetrahedral polyhedra are typical of chelate compounds containing the MN_4 coordination unit (M = Co, Ni, Cu).[17, 20, 24]

Most of complexes with the MN_2O_2 coordination unit (M = Ni, Cu) have planar structures with a *trans* arrangement of the ligands.[12, 20, 21, 24, 313–315] Palladium complexes are characterised exclusively by the square-planar coordination.[21] The tetrahedral coordination is observed in all beryllium complexes [317] and in most cobalt,[316] zinc, cadmium and mercury complexes.[285, 286] Chelate compounds of d^8 elements containing the MN_2S_2 coordination unit have *cis*-planar structures.[20, 22, 24, 25] These structural features of chelate azomethine complexes have been discussed in the literature many times, including from the viewpoints of the mutual effect of metals and ligands [21] and stereochemistry of metal chelates of the ML_2 type.[320]

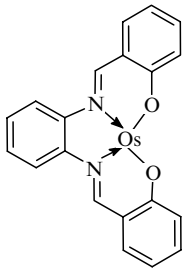
Most alkylene- or arylene-bridged chelate compounds with tetradentate bisazomethine ligands have planar tricyclic structures. In addition to the above-discussed bis(salicylidene)ethylenediamine chelates characterised by X-ray diffraction analysis, complexes **161**,[150] **162** [318] and **163** [319] also have analogous structures.



161: M = Ni, Cu.

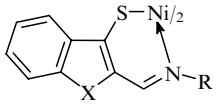


162



163

The coordination units in chelates of the MN_4, MN_2O_2 and MN_2S_2 types adopt a tetrahedral configuration as a result of annelation of heteroaromatic fragments to metallocycles.[20–22, 24] For example, these structures are typical of benzofuran- and pyrazole-based azomethine chelates (**164a** and **150**, respectively).[20]



164a,**b**: X = O (**a**), S (**b**); R = Bu^n, Ph.

A substantial flattening of the structure is observed on going from azomethines of the benzofuran series **164a** to azomethines of the benzothiophene series **164b**.[20]

When examining the dependence of the stereochemistry of azomethine chelate compounds of type **2** on the nature of the substituent R^4, the effects associated with the volume of this substituent or with the presence of additional donor centres are generally considered. The former effect causes primarily steric distortions of the polyhedra.[12, 21] The latter effect leads to an increase in the coordination number of complex-forming metal from 4 to 5 or 6.[22, 24, 25] Relying on these notions, the directed synthesis of complexes with the desired geometry of the coordination unit can be carried out.

Since the steric effects of the substituents at the nitrogen atom (R^4) have been discussed in sufficient detail,[12, 20, 21, 24] we will consider only some examples of these effects on the structures of azomethine chelates **2**.

In the crystalline state, nickel chelates **2** (M = Ni; R^4 = H, Alk, Ar) have square-planar or distorted tetrahedral configurations depending on the volume of the R^4 substituent,[21, 313] whereas the complex configurational equilibria including planar, tetrahedral and octahedral (intermolecularly associated) structures, were observed in solutions of these chelate compounds.[21] Distortions of the tetrahedral structures in copper chelates of type **2**, which are caused by substituents at the nitrogen atom exhibiting different steric effects, were unambiguously established by X-ray diffraction analysis. However, there is no consensus of opinion on the

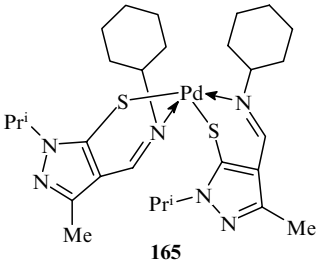
structures of these complexes in solution (*cf.* Ref. 21). It is believed that square-pyramidal or distorted tetrahedral configurations as well as square–tetrahedron equilibria can occur in solution.

The influence of the nature of the substituents at the nitrogen atom, which are able to form additional coordination bonds, on the structures of complexes **2** has been the subject of investigation over the past 40 years. The presence of these substituents leads primarily to an increase in the coordination number of the central atom in chelates **2**, *i.e.*, the coordination is changed from square-planar or tetrahedral to penta- or hexacoordinated. This fact allows one to perform the directed synthesis of chelate compounds of the azomethine series with pyramidal or octahedral structures.

In recent years,[101, 105–115] considerable attention has been given to the investigation of the structures of β-aminovinylimine chelates **48** containing the bulky diisopropylphenyl substituents R^3 at the nitrogen atom. In chelates with composition MLR^3, the access to complex-forming metals is sterically hindered.

Ligands containing the same substituents can form chelates of different stereochemical types.[22, 24, 215] In complexes of pyridine-containing azomethines, the nitrogen atom of the heterocycle can either form a bond with the metal atom (compound **108**[213]) or not (compound **107**[212]). The quinoline nitrogen atom is generally coordinated to the metal atom (compounds **111**[215, 216] and **112**[217]).

The dependence of the stereochemistry of chelate compounds on the nature of the metal atoms was partially considered at the beginning of this section. Noteworthy also is structure **165** characterised by the *trans* arrangement of the donor atoms and the 'umbrella' conformation of the coordination unit untypical of azomethine chelates of d^8 metals with the N,S-ligand environment.[292, 293] The formation of this structure is associated with several factors. In palladium complexes, the ligands generally exhibit the square-planar coordination mode. However, the pyrazole fragment and bulky cyclohexyl substituents at the nitrogen atoms induce structural distortions.

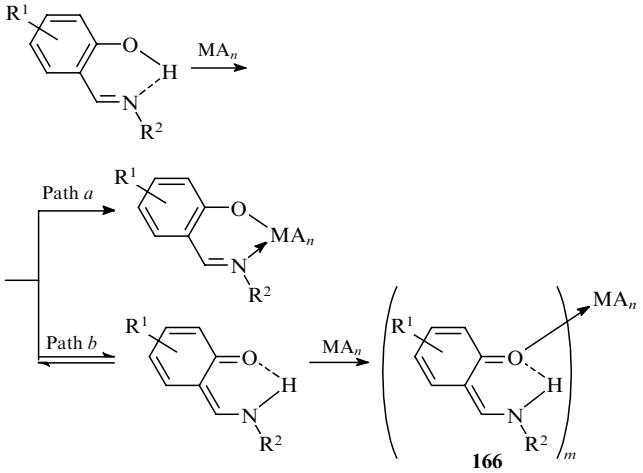


165

The structural analysis of nickel chelates with Schiff bases of aromatic and heterocyclic *o*-hydroxy(thio)aldehydes clearly showed the influence of the fine structure of ligand systems on the structures of azomethine metal chelates. Three coordination modes, *viz*., planar with a *cis* or *trans* arrangement of the ligands and tetrahedral, were found in the known complexes. The coordination mode is changed if the nature of the heteroatom in the chelate ring is changed, bulky substituents are inserted at the azomethine nitrogen atom, or on going from derivatives of aliphatic or aromatic aldehydes to heterocyclic analogues (Fig. 1).

III. Molecular complexes with azomethine ligands

o-Hydroxyazomethines serve as classical chelating ligands (Section II.2.b). However, as was first demonstrated in 1964,[321] these compounds can also be used for the preparation of molecular complexes **166**.[20, 22, 24, 28, 29, 322–325]



166

R = Alk, Ar; M = Cu, Zn, B, Al, Sn, Ti, Th; A = Hal, NO_3, NCS; m = 1, 2; n = 2–4.

The reaction path depends on the nature of the solvent.[323] Thus, metal chelates (Section II.2.b) are formed predominantly in media with high dielectric permeability (alcohols; path *a*), whereas molecular complexes **166** are generated primarily in aprotic non-polar media with low basicity (hydrocarbons, their halogen derivatives, ethers or mixtures of these solvents; path *b*).

Some reactions proceed in mixtures of benzene and alcohols. Thus, the direct reaction of the ligands with $ZnCl_2$ afforded complex **167**.[326]

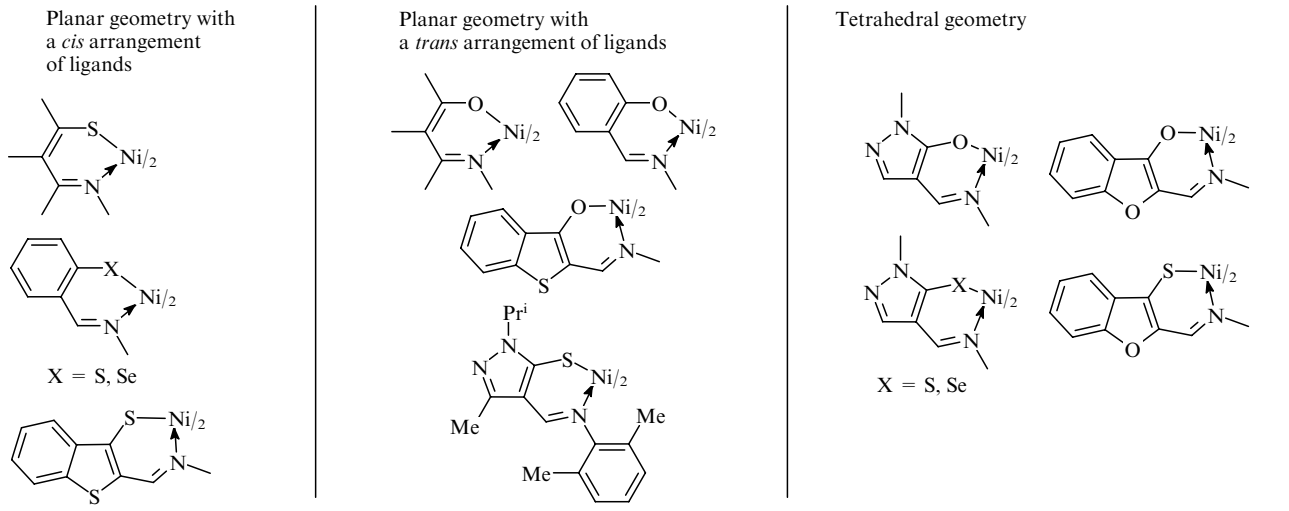
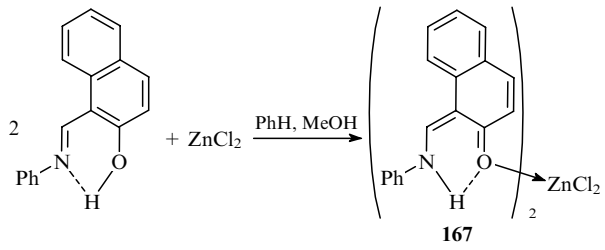
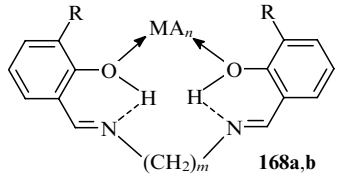


Figure 1. Coordination modes in nickel complexes.



167

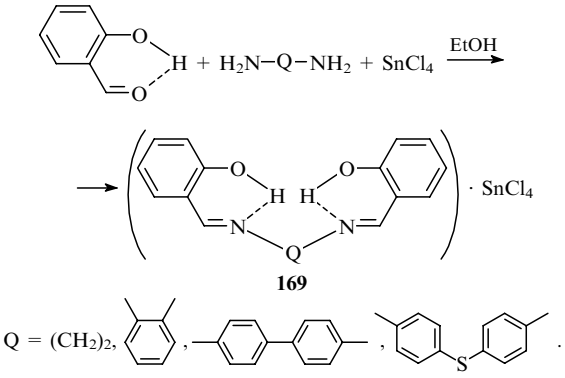
Bis(salicylidene)alkylenediamine molecular complexes **168a** [327] and **168b** [328] were synthesised analogously.



168a,**b**

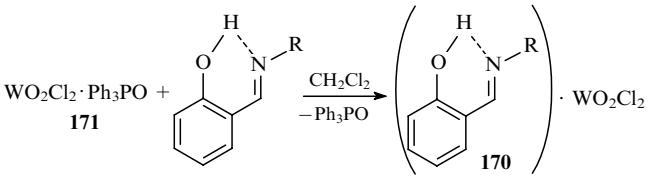
168a: R = H, M = Ca, A = NO_3, m = 3, n = 2;
168b: R = H, Me, OMe; M = Ti, Zr; A = F, Cl; m = 2, n = 4.

The template synthesis methods are employed more rarely. However, this approach was used for the preparation of adducts **169**.[329]



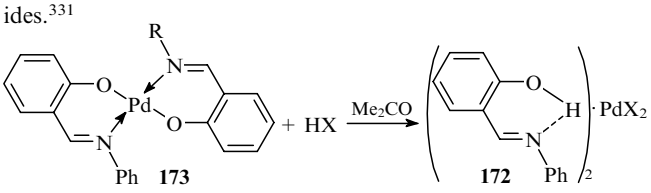
169

Molecular complex **170** was synthesised by ligand exchange in tungsten complex **171**.[330]



171 **170**

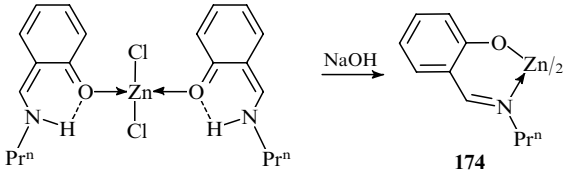
It was demonstrated that molecular complexes **172** could be prepared from chelates **173** under the action of hydrogen halides.[331]



173 **172**

X = Cl, Br, I.

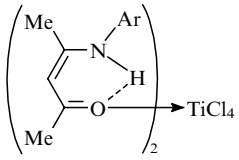
Recently,[332] metal chelates **174** were synthesised starting from a molecular complex analogous to complex **167**.



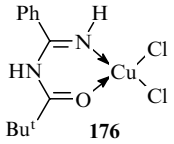
174

Taking into account the data on the synthesis of molecular complex **172**,[331] the possibility of the interconversion of chelates **2** and molecular complexes **3** may be considered as proved.

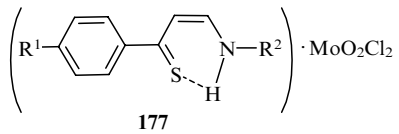
Among recent studies, let us mention the investigations of adducts of metal halides with β-aminovinyl ketones **175** [101, 333] and their analogues **176** [334] and **177**.[335]



175



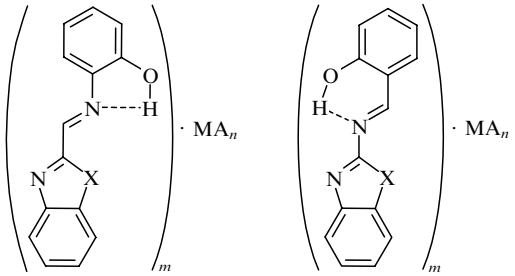
176



177

R^1 = H, Br; R^2 = C_7H_{15}, Bz, C_6H_4Me-4, C_6H_4OMe-4, C_6H_4Br-4.

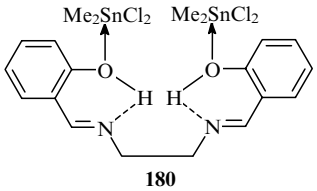
Molecular complexes **178** and **179** of metal halides with hydroxyazomethines containing five- or six-membered chelating fragments [336–338] were prepared by the reactions of the corresponding ligands with metal chlorides in anhydrous benzene (M = Sn, Ti) or methanol (M = Cu, Zn, Cd). Palladium complexes **178** and **179** were synthesised by the ligand-exchange reactions of Schiff bases with $PdCl_2(PhCN)_2$.



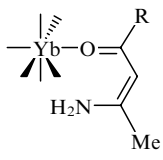
178 **179**

X = NR, O, S; M = Sn, Ti, Pd, Cu, Zn, Cd; m = 1, 2; n = 2, 4.

Several structural types were proposed for the molecular complexes under consideration.[20, 27, 29, 322–324] X-Ray diffraction studies demonstrated that hydroxyazomethines and β-aminovinyl ketones in molecular complexes **180** and **181a**,**b** act as monodentate ligands containing the O-donor centre. For the first time, this coordination mode was unambiguously established by X-ray diffraction analysis of complexes with bis(salicylidene)ethylenediamine **180** [339] and tris(acetylacetonato)(4-aminopent-3-en-2-one)ytterbium **181a**.[15, 340]

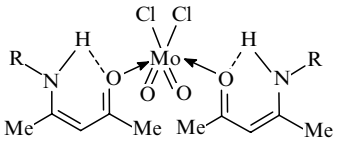


180

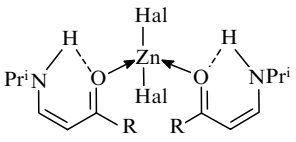


181: R = Me (**a**), Ph (**b**).

It is believed [339] that complexes **180**, like compound **168a**,[327] contain the ligand in the hydroxyimine form. However, X-ray diffraction analysis of complex **167**, which made it possible to locate the positions of protons in the structure, showed that the ligand occurs as the quinone-imine tautomer.[326] According to the results of X-ray diffraction analysis, the ligands in complexes **182** [341, 342] and **183** [343] also occur in the form of β-aminovinyl ketones.



182: R = Ph, C_7H_{15}.



183: R = Me, Pr^i.

It should be emphasised that IR spectroscopy cannot be employed for revealing the mode of location of the coordination bond and establishing the tautomeric form of the hydroxyazomethine or β-aminovinyl ketone ligands because the absorption bands corresponding to both types of coordination (at the nitrogen atom of the C=N bond or at the oxygen atom of the carbonyl group) have similar frequencies. The ^{1}H NMR spectra also do not provide conclusive information for the solution of these problems.[344]

The data surveyed in this section provide the basis for the directed stereochemical[24] and regioselective[24, 345] syntheses of azomethine metal chelates.

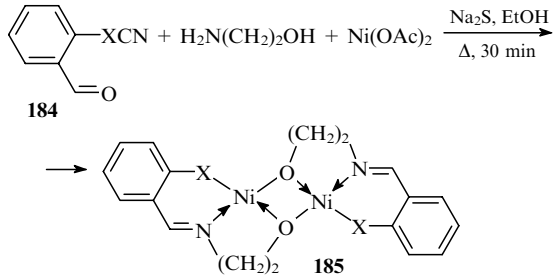
IV. Bi- and polynuclear azomethine complexes

Bi- and polynuclear complexes containing azomethine ligand systems are formed by the reactions of bi- and polydentate Schiff bases or their complexes as well as of polymeric azomethines with metal sources or upon polymerisation of monomeric metal complexes.

1. Bi- and polynuclear complexes of polydentate Schiff bases and their analogues

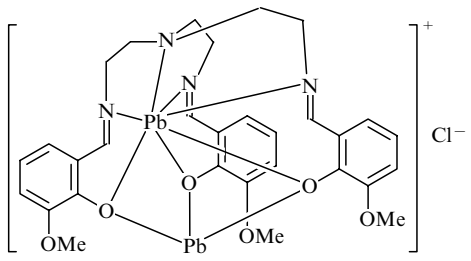
A series of chelating azomethines form not only mononuclear complexes but also bi- and polynuclear structures. Such structures were constructed and characterised for chelates prepared on the basis of bidentate Schiff bases of *o*-aminophenol **17**[64, 346] and its thio analogue **31**,[347] tridentate imino alcohols **83** (Y = OR) and iminothio alcohols **83** (Y = SR).[24, 177]

Reactions of substituted benzaldehydes **184** with aminoethanol and nickel acetate afforded binuclear chelates **185**.[348]

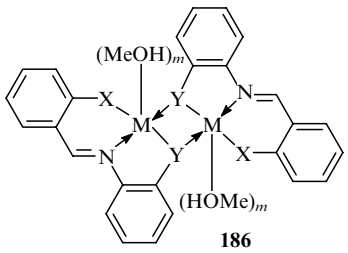


X = S, Se.

The lead complex with composition $[Pb_2L]Cl$ was prepared by the reaction of the tripodal $N(CH_2CH_2N{=}CH{-}C_6H_3OMe\text{-}3\text{-}OH\text{-}2)_3(H_3L)$ ligand with $PbCl_2$ in methanol in the presence of a base.[349]



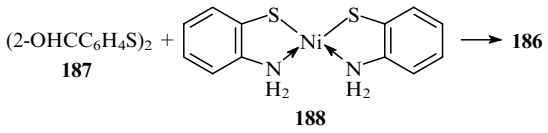
Binuclear complexes **186** with tridentate azomethine ligands[2, 24, 280, 350–353] were synthesised primarily from the ligands and the corresponding metal acetates.[350–353]



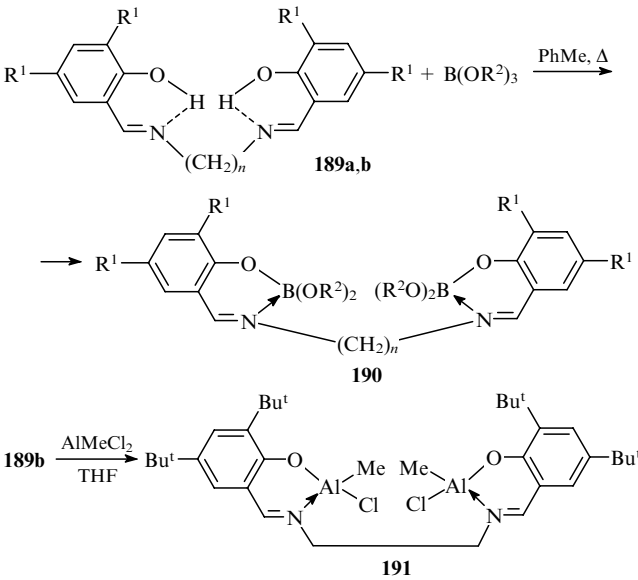
186

M = Co, Ni; X, Y = NTs, O, S; *m* = 0, 1, 2.

Binuclear nickel complex **186** (X = Y = S, M = Ni, *m* = 0) was prepared by the template method from disulfide **187** and complex **188**.[280]



Tetradentate azomethine ligand systems of the bis(salicylidene)ethylenediamine series[34, 218] form not only mono- but also binuclear chelates.[225, 251, 354–363] Such chelates were prepared for boron,[218, 357] aluminium,[218, 354, 359] gallium,[218] indium,[218] titanium,[358] europium and yttrium,[225, 357] cobalt,[355, 363] chromium and molybdenum,[34] iron,[356] nickel and copper,[361] and zinc.[251, 360] Complexes containing the bridging bis(salicylidene)alkylideneamino ligands **189a**,**b**, such as **190**[357] and **191**,[354] are the most widespread binuclear chelate compounds.



189a,**b**

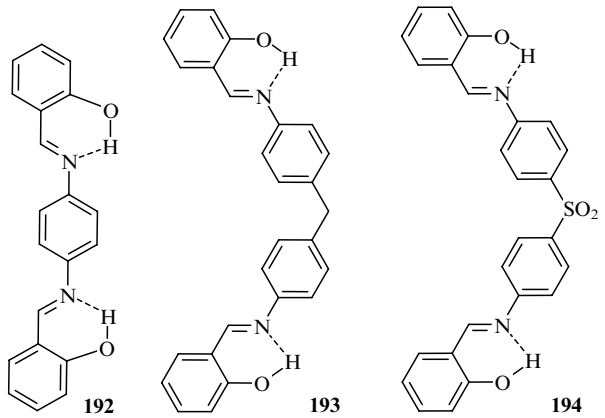
190

191

R^1 = H (**a**), Bu^t (**b**); R^2 = Me, Et; *n* = 2–6.

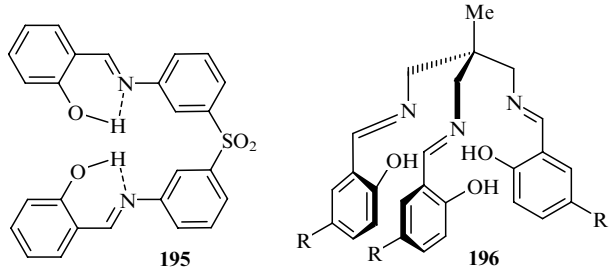
Conceivably, these binuclear structures are generated through the formation of intermolecular dimers due to coordination of the metal atoms by the oxygen atoms of the adjacent complex molecules.[34]

Bisbidentate Schiff bases **192**,[364] **193**,[365] **194**[365] and **195**[366] show promise for the construction of bi- and polynuclear azomethine complexes.



192 **193** **194**

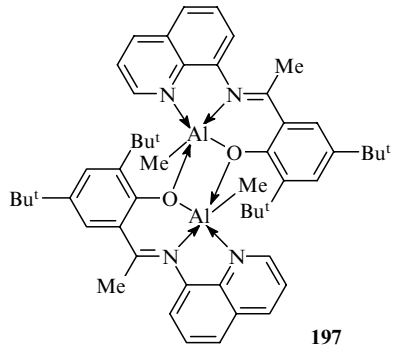
Binuclear structures were prepared on the basis of compounds **192**–**194**. Tetranuclear complexes were derived from ligand **195**. Tripodal hexadentate ligands **196** gave rise to bi- and trinuclear nickel complexes.[367]



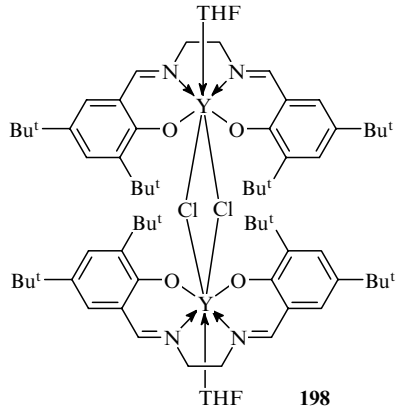
195 **196**

R = H, OMe.

Various bridging atoms, such as oxygen (for example, compound **197**[175]) or chlorine (compound **198**[368]), play an important role in the formation of binuclear structures.

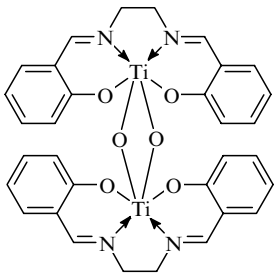


197



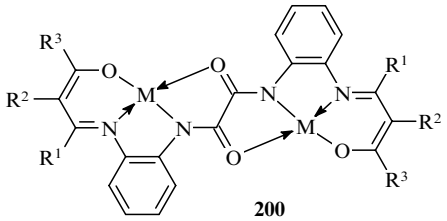
198

In some cases, atomic oxygen serves as an intermolecular bridge to form, for example, oxotitanium dimeric,[369–372] trimeric and tetrameric[370,373] complexes of tetradentate Schiff bases. Structure **199** provides an example of dimeric complexes.[372]



199

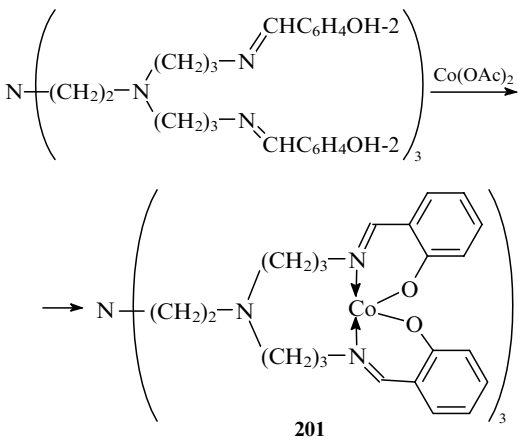
There are data on oxygen-bridged complexes derived from bidentate Schiff bases. For example, complexes of di-μ-oxodimanganese(IV) with *N*-alkyl-substituted salicylideneamines were synthesised.[374] Binuclear chelates **200**[375] formed by the complex polydentate ligand containing two β-ketoiminate fragments also belong to this type of complexes.



200

R^1 = H, Me; R^2 = H, COMe;
R^3 = Me, Ph, $C_6H_4NO_2$-4; M = Ni, Cu.

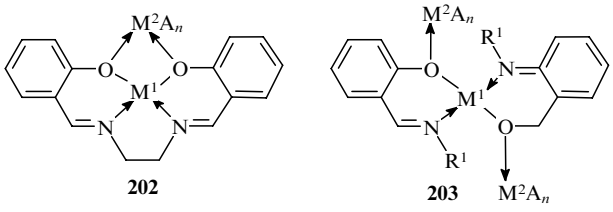
Trinuclear oxygen-bridged complexes **201**[376,377] were prepared based on dendrimers with salicylideneaminate groups.



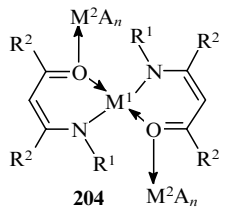
201

2. Bi- and polynuclear complexes with metal-containing azomethine ligands

In the 1960s, it was demonstrated[378–381] that the reactions of chelate compounds containing the azomethine ligands M^1L_m with metal salts M^2A_n can give rise to bi- (**202**[380,381]) and trinuclear (**203**[378] and **204**[379]) structures.



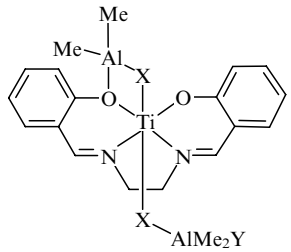
202 **203**



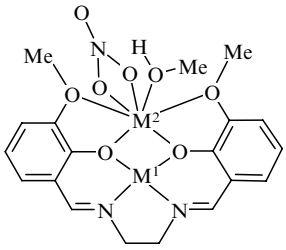
204

R^1 = Alk, Ar; M^1 = Ni, Cu; M^2 = Co, Ni, Cu, B, Al, Ti, Sn; A = Hal, NO_3.

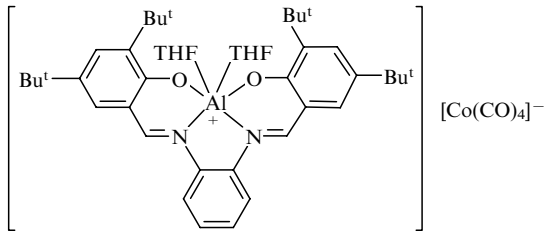
The direct reactions of ligands with sources of complex-forming metals were used for the preparation of complexes **205**,[358] **206**[382] and **207**.[383]



205: X = Cl, Br; Y = Me, Cl.



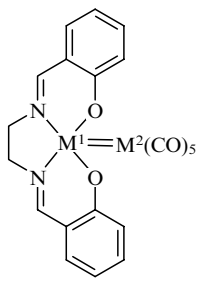
206: M^1 = Co, Ni, Cu, Zn; M^2 = Na, K, Cs.



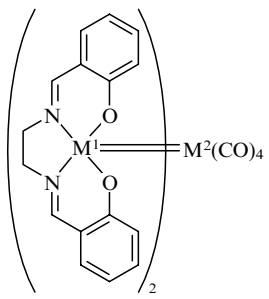
207

It should be noted that alkali salts in the heteronuclear structures under consideration can act as Lewis acids (M^2A_n). In particular, this is evidenced [382] by the formation of adducts **206** in the reactions of salicylaldimine complexes of divalent metals (Co, Ni, Cu, Zn) with sodium, potassium and cesium salts. β-Aminovinyliminate-based complexes **206** were described in the study.[384]

Unlike the above-considered binuclear halide complexes **202**, which were prepared by the reactions of the corresponding ligands with metal halides, coordination compounds of metal carbonyls were synthesised by ligand-exchange reactions.[385] These reactions afforded two types of complex compounds with the M^1-M^2 and $M^1-M^2-M^1$ bonds, *viz*., $M^1LM^2(CO)_5$ (**208**) and $(M^1L)_2M^2(CO)_4$ (**209**), respectively.



208

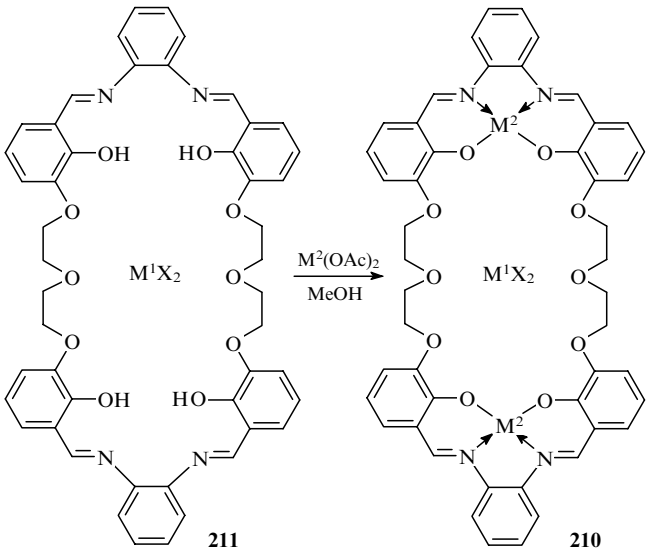


209

M^1 = Ge, Sn, Pb; M^2 = Cr, W.

Binuclear complexes can be prepared by the reactions of metal chelates, for example, of bis(salicylidene)ethyleneaminates or bis(tris)-β-diketonates, in chloroform.[386–391] In azomethine chelate compounds, Ni(II) [387–391] and Cu(II),[386, 389, 391] serve as the M^1 metal and hexafluoroacetylacetonates of Ba and Mg,[390] Y(III),[386–389, 391] La(III) [389, 391] and Gd(III) [386, 389, 391] act as metal salts (M^2A_n). The structures of these complexes were established by X-ray diffraction analysis. Like in complexes **202**, the coordinatively unsaturated oxygen atoms of the bis(salicylidene)ethylenediaminate anion are involved in coordination to lanthanides.

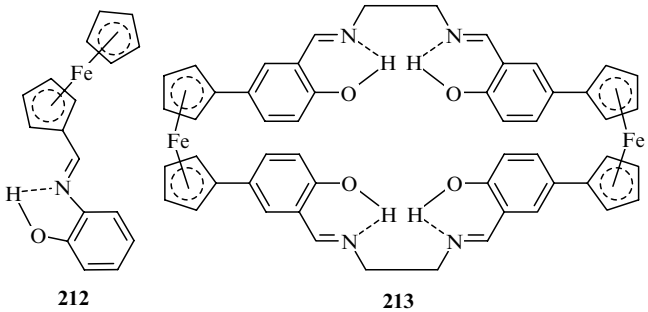
Bi- and trinuclear complexes were synthesised starting from coordination compounds of crown ethers containing the chelating hydroxyazomethine fragments.[392–394] As an example of these compounds, we refer to heterotrinuclear complex **210** generated from heterobinuclear complex **211**.[392]



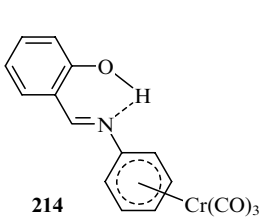
211 **210**

M^1 = $Ba(CF_3SO_3)_2$; M^2 = Cu, Ni.

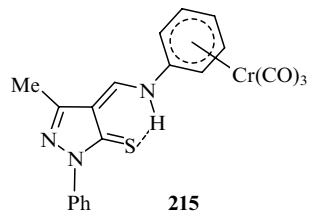
It should also be noted that Schiff bases containing organometallic fragments can be used as azomethine ligands. Ferrocene derivatives **212** [395, 396] and **213** [397] as well as derivatives of benzene chromium tricarbonyls **214** [398] and **215** [399] are examples of these Schiff bases.



212 **213**



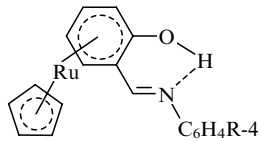
214



215

Ligands **212**, **213** and **214** give rise only to metal chelates, whereas azomethine **215** produces molecular complexes.

Ruthenium complex **216** shows promise as a ligand for the preparation of tri- and oligonuclear coordination compounds.[400] The former complexes can be synthesised by the reactions of ligand **216** with divalent metal acetates and the latter complexes can be prepared on the basis of metal salts in oxidation states 3 and higher.

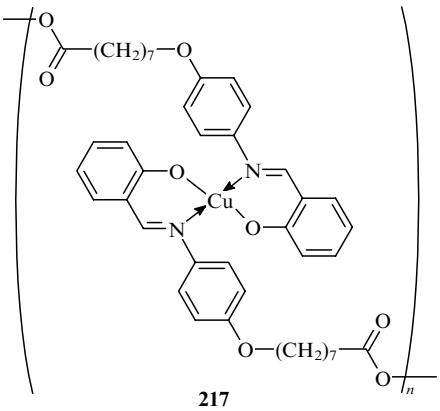


216

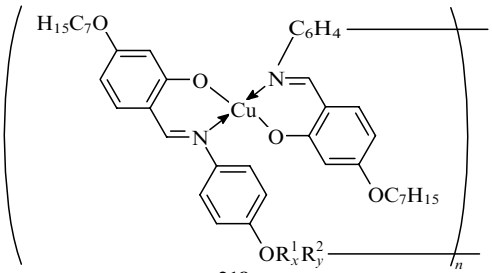
3. Azomethine metal polymers

Main methods for the preparation of polymers containing chelates of Schiff bases and their analogues involve the reactions with chelating macroligands and polymerisation of metal monomers.[31–33, 401–405]

Chelating polymeric azomethine ligands are used in synthetic reactions similar to those involving monomeric Schiff bases and their analogues (see Sections II and III). Thus, the reactions of the ligands with metal salts afforded polymers containing chelates (for example, **217**,[406] **218**[407]) and molecular complexes (**219**[405]). The metal exchange gave rise to metal polymers **220**.[32]

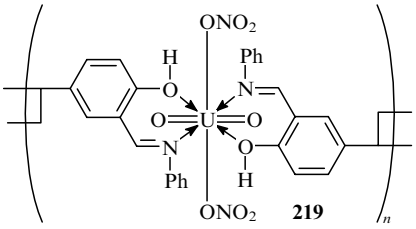


217

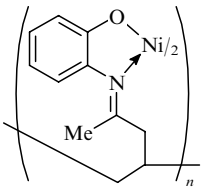


218

$R^1 = (CH_2)_{12}O$; $R^2 = (CH_2CH_2O)_3$; $x = 0-100$; $y = 100-x$.

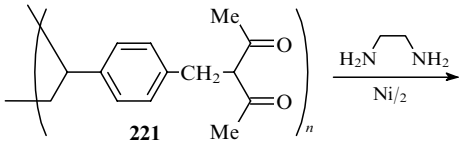


219

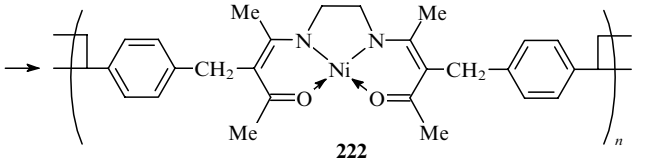


220

Polymer **222** was prepared from ligand **221** with the use of the template method.[32] This approach is particularly efficient when metal polymers cannot be prepared by the reactions of ligands with metal salts or through metal exchange.

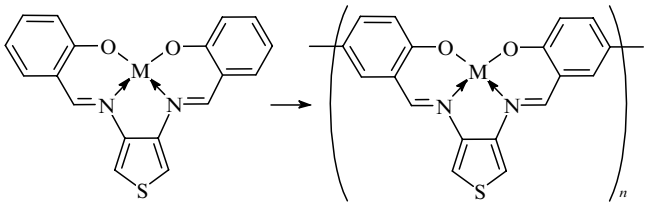


221

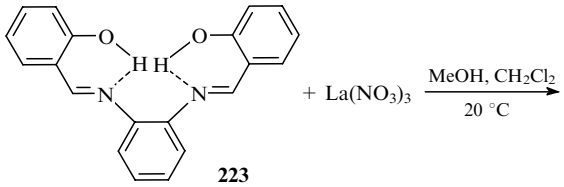


222

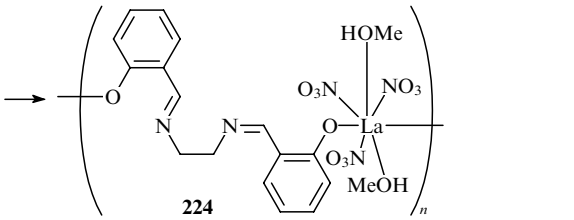
The oxidative polymerisation of monomeric chelates was used for the preparation of polymers with bissalicylidene derivatives of aromatic[408, 409] or heterocyclic[410, 411] amines.



In some cases, polymeric azomethine complexes can be prepared in the course of usual syntheses. In particular, the reaction of ligand **223** with La(III) nitrate afforded polymeric structure **224** containing fragments of molecular complexes with the bis(salicylidene)ethylenediaminate ligand coordinated through the oxygen atom.[412]



223



224

Polymeric complexes of Schiff bases can also involve bridging fragments, for example, oxygen[413] or metal chelates.[414]

V. Conclusion

The available data on metal complex compounds with azomethine ligands make it clear that the employment of the techniques of rational design in the search for ligand systems and synthesis procedures can be a help in performing the directed synthesis of complexes in which the mode of coordination and geometry of metallocycles are prespecified.

In addition to synthetic and structural problems arising in research on metal coordination compounds with azomethine ligands, theoretical and applied aspects are also of great importance, including competitive coordination,[24, 27] standard and non-standard binding of metals to chelating ligands,[28, 29] stabilisation through the complex formation of tautomeric forms untypical of free ligand systems[77] and isomerism of coordination units within chelates.[24, 27]

Complexes of Schiff bases and their structural analogues are of practical importance because they allow one to perform the directed design of new types of azomethine coordination com-

pounds exhibiting catalytic, magnetically anomalous and liquid-crystalline properties. In this connection, it should be emphasised that metal chelates of Schiff bases in which the complex-forming metal atom is shielded by bulky substituents (compounds of type **48**: $R^1 = C_6H_3Pr^i_2$), belong to new-generation catalysts for polymerisation, copolymerisation, enantioselective synthesis and oxidation of alkenes (predominantly, epoxidation). In addition to the studies cited in the present review [101, 105–136] these problems have also been considered in the studies devoted to the same subject.[415–417]

Bi- and trinuclear complexes of type **204** (A = Hal; see also Ref. 418) serve as efficient catalysts for oligo- and polymerisation as well as for hydrogenation. These complexes, like binuclear chelates **202** (MA_n are lanthanide tris-hexafluoroacetylacetonates),[386, 419] and azomethine coordination compounds with azole free radicals **89** and **90** (molecular ferromagnetics) [420] exhibit magnetically anomalous properties.

Data on azomethine metal complexes possessing liquid-crystalline properties have been surveyed in the review [421] and more recent publications.[422, 423]

Taking into account considerable advances that have been made in the construction of biomimetic models of active sites of non-haeme metalloproteins on the basis of azomethine metal chelates,[267, 295, 299, 307, 424, 425] this line of studies shows considerable promise.

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