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CHEMISTRY OF POLY(AZOLYL)BORATE COMPLEXES

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ABSTARCT

A few poly(azolyl)borate complexes with azolyl groups other than pyrazole have been investigated, although it has been shown that the chemistry of poly(azolyl)borate complexes may be critically dependent upon the pattern of ring substitution. This aspect has induced several researchers to investigate the behavior of metal complexes of analogous ligand systems with modified steric and/or electronic properties, such as poly(tetrazolyl)borate, poly(1,2,3-benzotriazolyl)borate, poly(2-sulfanyl-1-ethylimidazolyl)borate and poly(imidazolyl)borate ligands. These modifications have led to new scorpionates capable of bridging different metal centers, yielding dimers or coordination polymers characterized by peculiar spectroscopic and structural features. Some of these new ligands are very promising for the synthesis of inorganic materials with inner cavities useful for the catalysis of organic reactions or with collective magnetic phenomena for the design of molecular-based ferro magnets.

KEYWORDS: Poly(Tetrazolyl)Borate, Poly(Imidazolyl)Borate Ligands

Two features of triazole-based ligands make them attractive candidates for further examination: (i) they should be electron-withdrawing relative to their pyrazole-based counterparts so that the properties of metal complexes differ accordingly (Reedi Marzano *et al.*, 2006); (ii) the exo ring-nitrogen atoms of triazole-based ligands may bridge between metal centers, thereby creating coordination polymers with interesting solid-state structures and optical properties, as recently shown by Janiak *et al.*, (1996). Moreover, the exocyclic nitrogen atoms might take part in hydrogen-bond interactions assisting the formation of two-dimensional water-intercalate or water-layer clathrates (Jarvis, 1962), thus leading to water soluble species (Janiak, 2000).

AZOLYL BORATE

Giancarlo Gioia Lobbia *et al* have recently initiated an investigation into the structural and spectroscopic properties of mixed phosphane/ N-donor derivatives of copper(I) (Lobbia *et al.*, 1997), silver(I) and gold(I) (Lobbia *et al.*, 2005; Macleod *et al.*, 1996; Trofimenko *et al.*, 1987; Heuer *et al.*, 2000; Edelmann, 2001; Macleod *et al.*, 1996; Hanson 1999). They have been interested in complexes incorporating poly(azolyl)borate and phosphane ligands with different steric and electronic properties. The different coordination characteristics of poly (triazolyl)borate species offer the prospect of new and interesting chemistry, paralleling that of pyrazolyl and imidazolyl arrays (Hanson, 1999). In their research concerning modifications of the poly(pyrazolyl)borate ligands in which the pyrazolyl rings are replaced by 1,2,4-triazolyl or imidazolyl rings, some interesting single stranded silver(I) coordination polymers containing bridging poly(1-imidazolyl) borates have been reported (Navon *et al.*, 2000). The substitution of triazole for pyrazole in poly(pyrazolyl)alkanes to form poly(triazolyl)alkanes as bis(1,2,4-triazolyl)alkane ligand extends the capability to coordinate silver(I) salts through the additional nitrogen atoms, yielding two- and three-dimensional coordination polymers (Fujita *et al.*, 1994). e.g.; dihydrobis(1,2,4-triazolyl) borate, $[H2B(tz)2]^-$ hydrotris(1,2,4-triazolyl)borate, $[HB(tz)_3]^-$.

Although a cobalt complex of hydrotris(1,2,4-triazolyl)borate was reported by Trofimenko in 1967 (Ibrahim *et al.*, 1994), the chemistry of triazolylborate ligands has remained undeveloped. Two features of triazole-based ligands make them attractive candidates for further examination: i) they should be electron-withdrawing relative to their pyrazole-based counterparts and the properties of metal complexes should differ accordingly (De Munno *et al.*, 1994); ii) the exo ringnitrogen atoms of triazole-based ligands may bridge between metal centers, thereby creating coordination polymers with interesting solid-state structures and optical properties (Xiao *et al.*, 1994) Moreover, the exocyclic nitrogen atoms might take part in hydrogen-bond interactions the formation of two-dimensional water-intercalation or water layer clathrates, (Macleod *et al.*, 1996) thus leading to water soluble species. (Shiu *et al.*, 1993) An investigation into the structural and spectroscopic properties of mixed phosphine/N-donor derivatives of copper(I), silver(I) and gold(I) has been

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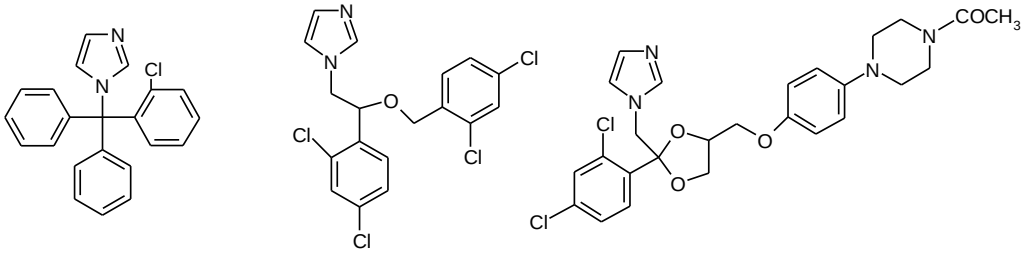
initiated (Calabrese *et al.*, 1986). As a part of this study they have explored complexes incorporating poly(azolyl)borate and phosphine ligands with different steric and electronic profiles. The different coordination characteristics of poly(triazolyl)borate species offer the prospect of new and interesting chemistry, paralleling that of pyrazolyl, benzotriazolyl and imidazolyl arrays (Trofimenko *et al.*, 1987). They have synthesized, some new metal complexes obtained from the interaction of dihydrobis(1,2,4-triazolyl)borate $[H_2B(tz)_2]^-$ and tris(1,2,4-triazolyl)borate $[HB(tz)_3]^-$ with CuCl and tertiary monophosphines.

Polynuclear co-ordination compounds containing derivatives of 1,2,4-triazole have been of great interest during last decade. Several studies have dealt with search for magneto-structural correlations for transition metal (II) compounds containing N^1, N^2-1,2,4-triazole bridges. On the other hand polynuclear iron(II) 1,2,4-triazole compounds have been found to yield spin-crossover materials exhibiting co-operative behavior. In order to acquire a more detailed understanding about the propagation of the magnetic super exchange via diatomic N-N bridges, the structure and magnetic properties of doubly N^1, N^2-1,2,4-triazole bridged binuclear Cu (II) compounds have been investigated. These compounds contain polyfunctional 1,2,4- triazole derivatives with N-donating substituents forming five membered chelate rings. The Cu(II) ions are linked in equatorial co-ordination plane by two N^1, N^2 bridging 1,2,4 triazole ligands. For the class of compounds the magneto-structural co-relations could be established for the first time. In the compounds the magnetic exchange is propagated via the $d_{(x^2-y^2)}$ orbitals on Cu(II) ions which interact with the orbitals of the nitrogen atoms of the bridging ligand. It has been experimentally found that the maximal value for the isotopic interaction J is attained, when the 1,2,4-triazole ligands like the Cu(II) ions in most symmetrical way allowing the N-Cu-N angles to be close to 90°. An increase in these N-Cu-N angles, which in most cases is accompanied by asymmetric bridging of μ- N^1,N^2-1,2,4-triazole ligands leads to a decrease in value of J. Therefore the dependence of the J-value on structural parameters has a certain relation found by not filled and nodgson for planar dihydroxo-bridged Cu(II) compounds.

PHARMACUTICAL USES OF AZOLE

Azoles are a class of five-membered nitrogen heterocyclic ring compounds containing at least one other noncarbon atom, nitrogen, sulfur or oxygen. The parent compounds are aromatic and have two double bonds, there are successively reduced analoges (azolines and azolidines) with less number of double bonds. Azoles maintain the prefix upon reduction such as pyrazoline, pyrazolidine, except for pyrrole, which has no azole suffix and is reduced to pyrroline and pyrrolidine. The azoles include pyrazoles, imidazoles, triazoles and tetrazoles.

Azoles with oxygen atom(s) in the ring are known as oxazole, isozazole and dioxazole and with sulphur atom in the ring are thiazoles and isothiazoles. Azoles have long been targeted for synthetic investigation because of their known biological properties like cognitive-enhancing and anxiolytic like activity (Clements-Jewery *et al.*, 1988; Roppe *et al.*, 2004; Geronikaki *et al.*, 2004; Hashimoto *et al.*, 2010). The antifungal agents of azoles (Fig. 1 {1 to 6}) are useful drugs and are widely used for the treatment of topic or inner mycoses, in particular AIDS-related mycotic pathologies (Sheehan *et al.*, 1999; Navon *et al.*, 2000; Ablordeppey *et al.*, 1999; Santini *et al.*, 1998).



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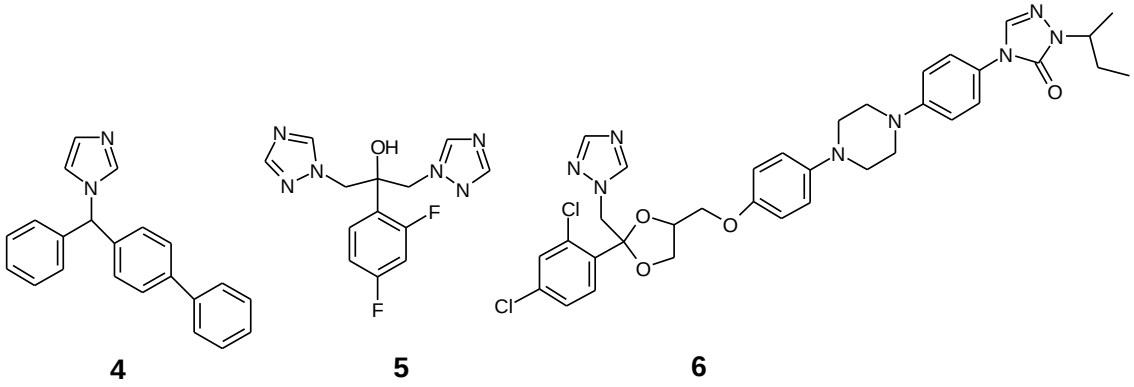
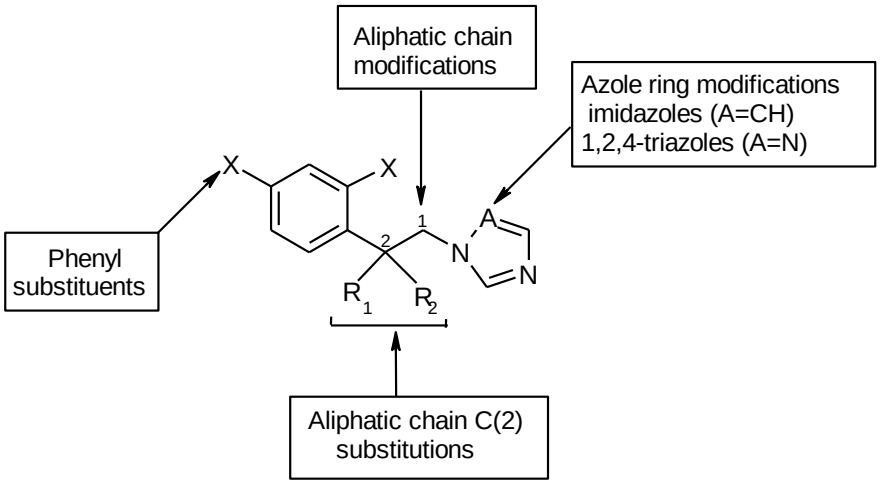


Figure 1:

It is well-known that azole derivatives block ergosterol biosynthesis, causing its depletion and accumulation of lanosterol and some other 14-methylsterols (Santini *et al.*, 1998; Santini *et al.*, 1998; Effendy *et al.*, 1998). Such sterols alter membrane fluidity with concomitant reduction in the activity of membrane-associated enzymes, increased permeability, and inhibition of cell growth and replication (Lobbia *et al.*, 2001). Imidazoles representing one class of antifungal azole derivatives have shown a broad spectrum of antifungal activities both *in vitro* and *in vivo* (Pellei *et al.*, 2000; Lobbia *et al.*, 2004). The development of imidazoline compounds has contributed significantly to the therapy of both superficial and systemic mycotic infections. Numerous studies on the structure-activity relationships (SAR) of antifungal azoles have been developed since the discovery of the first imidazoles, and these studies have led to new compounds endowed with better biological and /or pharmacological properties. The principal structural modifications introduced by the structural activity relations (SAR) in the field of imidazole antifungal agents have been summarized in (Fig. 2).



X= Cl, F; A= CH,N

R_1=R, OR, Ar R_2=H

R_1=R_2=1,3-spirodioxolane ring, =N-OR

Figure 2:

The SAR studies reveal the presence 2,4-dihalophenyl ring linked by an ethane chain to a nitrogen of an azole ring CH, or triazole. The ethane chain is often substituted on its C(2) by ethereal groups, as in miconazole or in econazole , by the 1,3-spirodioxolane ring, as in ketoconazole (Fig. 3) or in itraconazole, or again, oximethereal groups may be present in the same position as in oxiconazole (Lobbia *et al.*, 2002).

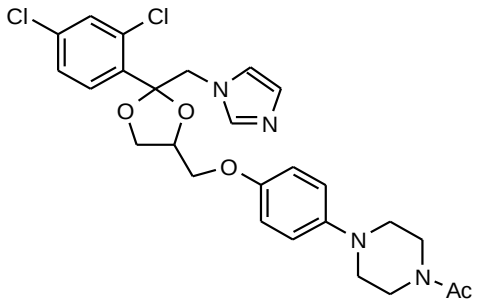


Figure 3:

Due to the considerable fact, that azoles show a number of biological importance, a number of researchers have tried to develope several different series of azoles such as diazole, triazole, oxazoles, dioxazoles and thiazole.

During recent years thiadiazole compounds, have been investigated for their biological properties such as antimicrobial (Farghaly *et al.*, 2012), antituberculosis (Oruç *et al.*, 204), antinflammatory (Schenone *et al.*, 2006; Kadi *et al.*, 2010), anticonvulsant (Sharma *et al.*, 2013), antihypertensive (Dias and Lu, 1995), local anesthetic (Dias and Wang, 2000), anticancer (Rheingold *et al.*, 2000), and hypoglycemic activities (Siemer *et al.*, 2001). The treatment of mycobacterial infections, especially tuberculosis has become an important problem due to the emergence of multidrug-resistance. A thiadiazole derivative, 2-(4-chloropheny-lamino)-5-(4-aminophenyl)-1,3,4-thiadiazole, showed 57% inhibition against *Mycobacterium tuberculosis* (Karakuş and Rollas, 2002). This observation led many of the researchers to search for some new thiadiazoles with pote antituberculosis activity. A series of 2,5-disubstituted-1,3,4-thiadiazoles were synthesized (Han *et al.*, 1989) and the compounds have been elucidated and screened for the antituberculosis activity against *Mycobacterium tuberculosis*. Among these compounds 2-phenylamino-5-(4-fluorophenyl) -1,3,4-thiadiazole (Fig. 4) showed the highest inhibitory activity.

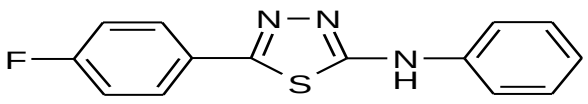


Figure 4

Antimycin A1 (Fig. 5), a dilactone salicylamide, was isolated from *Streptomycin sp.* (Bolgunas *et al.*, 2006), is a potent inhibitor of the Qi site of complex III (*bc*1 complex) in the mitochondrial respiratory chain, with a dissociation constant with bovine heart mitochondrial particles of 32 pM (Polak, 1982). During the binding mode studies, it was found that antimycin forms extensive hydrogen bonding network to the enzyme Lys 227 and Asp 228 considering the role of formamide in binding. The azole-fused salicylamides were prepared (Imramovsky *et al.*, 2011) as analogues of antimycin and assayed for activity at complex III of the mitochondrial respiratory chain. The activity of 7-hydroxyindazole-6-carboxylic Acid, 4-(4-trifluoromethylphenoxy)phenylamide approached that of antimycin in inhibitory potency and showed growth reduction of *Septoria nodorum in vitro*.

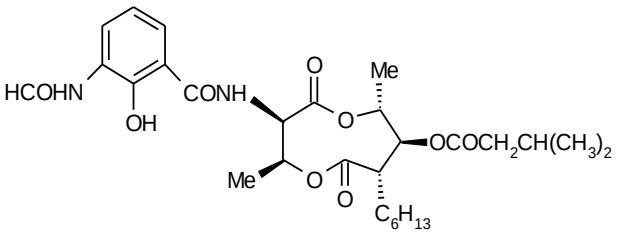


Figure 5

Oxazoles an important member of the azole family also contains a number of biologically active molecules, which play an important role in the drug chemistry. A number of compounds had been screened for antituberculosis agents including dihydrophinazines (Rossello *et al.*, 2002; Desai and Baxi, 1992; Gawande and Shingare, 1987; Mamolo *et al.*, 1996), indoles and ureas (Velezheva *et al.*, 2004; Mullican *et al.*, 1993; Oruç *et al.*, 2004). However oxazoles were always focused because of their inhibitory activity against mycolic acid biosynthesis. A series of optically active 6-nitro-2,3-dihydroimidazo[2,1-*b*]oxazoles substituted at the 2-position with various phenoxymethyl groups and a methyl group (Fig. 6), were synthesized (Sasaki *et al.*, 2006) and their efficiency were screened against both drug-susceptible and drug-resistant strains of *Mycobacterium tuberculosis*, using *in vitro* and *in vivo* methods.

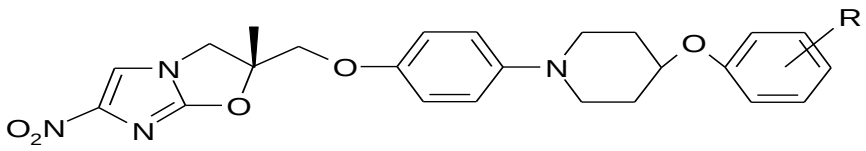


Figure 6

Screening of this novel series of (*R*)-form optically active 6-nitro-2,3-dihydro-imidazo[2,1-*b*]oxazole derivatives for *in vitro* antituberculosis activity and *in vivo* oral efficacy indicated that compounds with substituted phenoxymethyl groups and a methyl group at the 2-position are a new class of agents endowed with highly potent antituberculosis activity (Von Jagow and Link, 1986; Bolgunas *et al.*, 2006; Kikuchi *et al.*, 1997).

Oxybenzylglycine (Fig. 7), possing an oxazoline group, has been identified as a non-thiazolidinedione PPAR R/ç dual agonist that shows potent activity *in vitro* at human PPARR (EC_{50} = 320 nM) and PPARç (EC_{50} = 110 nM). It shows excellent efficacy for lowering glucose, insulin, triglycerides, and free fatty acids in genetically obese, severely diabetic *db*/*db* mice and has a favorable ADME profile. Oxybenzylglycine is currently in clinical development for the treatment of type 2 diabetes and dyslipidemia (Devasthale *et al.*, 2005).

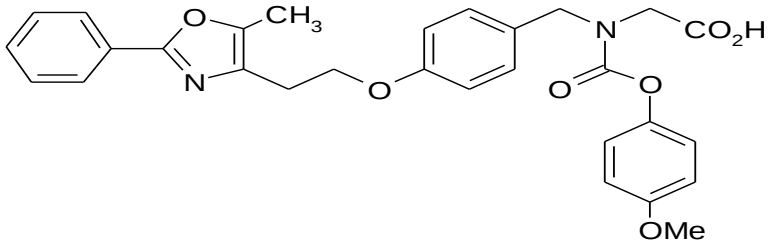


Figure 7

Considering the pharmacological importance of azole family and importance of 1,2,4-triazoles as being widely used in pharmaceuticals and agriculture chemicals, which may be connected to their similarity in geometry as well as coordinating properties. We have synthesized three different boron-triazole compounds and incorporated metal in them. The main aim of the research was to find a new drug, which can show better antimicrobial activity. The ligands prepared were screened for the same purpose, but to our unfortunate the metal complexes formed were neither soluble in any of the organic solvents nor in water. So it is not possible to screen these compounds for the antimicrobial activity. To our knowledge it was the first attempt to consider this class of complexes for the antimicrobial purpose.

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