**DIPYRROMETHANE, ITS DERIVATIVES AND THEIR METAL COMPLEX: APPLICATIONS AS CHEMOSENSORS AND BIOLOGICAL ACTIVITY**

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**Introduction**

Pyrrole ([C](https://en.wikipedia.org/wiki/Carbon)4[H](https://en.wikipedia.org/wiki/Hydrogen)4[N](https://en.wikipedia.org/wiki/Nitrogen)H) is a very important five membered heterocyclic aromatic organic compound having five-membered ring [1]. Pyrrole is firstly obtained by the dry distillation of protein by **Rung** in 1834. Pyrrole undergoes electrophilic aromatic substitution predominantly at the 2 and 5 positions instead of 3 and 4 as given in **Figure 1**.



**Figure 1**: Resonating structures of pyrrole

Pyrrole nucleus occurs in many natural compounds and it is a biosynthetic precursor to many natural products such as porphyrins- heme, chlorins and chlorophylls in **Figure 2**. The pyrrole-hydrazone and their metal complexes may show different biological activities such as antituberculosis and antimicrobial and used as versatile starting material for further synthesis of various types of organic compounds. Hydrazide-hydrazones having an azomethine proton –NH-N=CH- constitute an important class of compound for new drug development. They are mainly used as antitubercular, antimicrobial agent and potentially DNA damaging and mutagenic agents due to presence of >N-N=C< functional frame. They have strong coordinating ability towards different metal ions [2-5].





**Figure 2**: Natural products containing porphyrin ring

**Dipyrromethanes**

Dipyrromethanes are fully conjugated, planar bipyrrolic units that have attracted recent attention as ligands in supramolecular self-assembly. It covers a broad range of compounds with wide spectrum of activity. They have great potential application in various areas such as chemosensors, photo–induced energy, electron transfer, molecular based memory storage, small molecular activation, multi-electron redox catalysis, molecular devices. They are the most suitable starting materials for the total synthesis of pyrrole containing macromolecules, calix[n]pyrroles, porphyrin and boron-dipyrrin dyes. Derivative of dipyrromethanes is also potentially attractive structures for the development of new optical anion sensors, for application in biological systems and in the settling of environmental problems [6]. The general structure of Dipyrromethane is given in **Figure 3**.

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**Figure 3**: Structure of Dipyrromethane

**Dipyrromethanes types**

The compounds which possess two pyrrole units linked direct to each other or via linkers. Dipyrromethanes play the key role in photo-physical and redox processes in nature. Depending on nature of connecting linkers dipyrroles have been classified (**Figure 4**) in the following categories such as:



**Figure 4**: Classification of dipyrromethane

**Dipyrromethanes derivatives**

Dipyrrolic compounds in which two pyrrole units are linked via *meso*-carbon of (>CH2 or >CH->C<) as linker. For simplicity, they are categorized as:-

*1: Meso-unsubstituted dipyrromethanes.*

*2: Meso-substituted dipyrromethanes.*

*3: Symmetrical dipyrromethanes.*

*4: Asymmetrical dipyrromethanes.*

**1**: ***Meso*-unsubstituted dipyrromethanes**

**Table 1** shows various types of synthesized and natural *meso*-unsubstituted dipyrromethanes.

|  |  |
| --- | --- |
| **Table 1**: Shows various *meso*-unsubstituted dipyrromethanes derivatives. | |
| (a) 5-Unsubstituted dipyrromethane derived from ethyl tetrahydroisoindole-2-carboxylate and dimethoxymethane. | [7] |
| (b) Pyridinium salt of 2-bromomethylpyrroles reacts with lithium salts of pyrrole-2-carboxylic acids in polar solvents. | [8] |

**2**: ***Meso*-substituted dipyrromethanes**

Synthesis of *meso*-substituted dipyrromethanes can be of two categories: mono and disubstituted. They are as follows:-

(**a**) ***Meso*-monosubstituted dipyrromethanes**

*Meso-*monosubstituted dipyrromethanes is shown in **Table 2**.

|  |  |
| --- | --- |
| **Table 2**: Shows *meso*-monosubstituted dipyrromethane derivatives. | |
| (a) From vinyl pyrrole in the presence of TFA or InCl3 as catalyst.      (b) 2, 2’-substituted dipyrromethanes with EtMgBr and benzoyl pyridenyl sulfide. | [9] |
| [10] |
| (c) 2, 2’-bis (amido) and *meso*-substituted dipyrromethanes. | [11] |
| (d) Alkylthio unit is α-pyrrole protecting group. | [12] |
| (e) *Meso*-substituted dipyrromethanes in the presence of non-toxic CAN catalytic at room temperature. | [13] |
| (f) In *meso*-substituted dipyrromethanes, H2SO4.SiO2 to be used.    (g) Using molecular iodine under acetic acid. | [14]  [15, 16] |
| (h) Using CF3CO2H and CH3SO3H. | [17] |
| 1. Imidazolyl-dipyrromethane. | [18] |

(**b**) ***Meso*-disubstituted dipyrromethanes**

*Meso-*disubstituted dipyrromethanes are given in **Table 3**.

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| --- | --- |
| **Table 3**: Shows *meso*-disubstituted dipyrromethane derivatives. | |
| (a) Synthesis of *meso*-disubstituted dipyrromethanes in the presence of BF3.Et2O and EtOH as catalyst. | [19] |
| (b) *Meso*-disubstituted dipyrromethane with water in the presence of novel acid as catalyst at room temperature. | [20] |
| (c) In a pestle and mortar, mixture of pyrrole, ketone and I2 was crushed at room temperature. Various catalysts to be used for the synthesis of dipyrromethane such as TiCl4, TFA, pyrrolidinium tetrafluoroborate, *p*-toluenesulfonic acid.    (d) *Meso*-disubstituted dipyrromethane can also be catalyzed by a dinuclear ruthenium complex, Ru2(CO)4(PPh3)2Br4. | [21-23]    [24] |

**3**: **Symmetrical dipyrromethanes**

Symmetrical dipyrromethane means that both sides are identical and both part matches exactly when one half is like an image of the other half in a mirror. Various types of symmetrical dipyrromethanes are shown in **Table 4**.

|  |  |  |
| --- | --- | --- |
| **Table 4**: Shows symmetrical dipyrromethane derivatives. | | |
| (a) Benzyl 4-(2-methoxycarbonylethyl)-3, 5-dimethylpyrrole-2-carboxylate with bromine in diethyl ether. | [25] | |
| (b) Bromination of α-methylpyrrole in AcOH/AcONa affords α-acetoxymethylpyrrole which undergoes self-condensation in MeOH and HCl to give dipyrromethane.    (c) 2-Unsubstituted cyanovinyl pyrrole in the presence of BF3.Et2O as catalyst. | | [26]    [27] |

**4**: **Asymmetrical dipyrromethanes**

Asymmetrical dipyrromethane means that both sides are non-identical in some way. Various types of asymmetrical dipyrromethanes are shown in **Table 5**.

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| --- | --- |
| **Table 5**: Shows asymmetrical dipyrromethane derivatives. | |
| (a) Asymmetrical dipyrrolylmethane (TFA used as a catalyst). | [28] |
| (b) 5-triﬂuoromethyl-substituted dipyrromethanes derived in the presence of P2O5. | [29] |

**Dipyrromethene**

2,2'-Dipyrromethene is also called Dipyrromethene or dipyrrin with chemical formula C9H8N2 whose skeleton can be described as two [pyrrole](https://en.wikipedia.org/wiki/Pyrrole) rings C5N connected by a [methyne bridge](https://en.wikipedia.org/wiki/Methyne) =CH- through their [nitrogen](https://en.wikipedia.org/wiki/Nitrogen)-adjacent (position-2) carbons; the remaining bonds being satisfied by [hydrogen](https://en.wikipedia.org/wiki/Hydrogen) atoms. It is an unstable compound that is readily attacked by [nucleophilic](https://en.wikipedia.org/wiki/Nucleophyl) compounds above -40 °C.

**Synthesis**

2, 2'-Dipyrromethene can be prepared by [oxidation](https://en.wikipedia.org/wiki/Oxidation) of [2, 2'-Dipyrrolemethane](https://en.wikipedia.org/w/index.php?title=2,2%27-dipyrrolemethane&action=edit&redlink=1) with DDQ at -78 °C in dry DCM solution [30] is shown in **Scheme 1**.



**Scheme 1**: Synthesis of 2, 2'-Dipyrromethene

**Reduction of Dipyrromethene**

Reduction of Dipyrromethene with sodium borohydride also furnishes Dipyrromethane, and establishes that Dipyrromethane and Dipyrromethene are fully interconvertible in the synthetic sense is shown in **Scheme 2**.



**Scheme 2**: Reduction of Dipyrromethene

**Applications**

**a. Molecular probes and dyes**

Dipyrromethanes are used as versatile ligands for the syntheses of organic framework of BODIPY [31, 32], which are used extensively as molecular probes and dyes. The general route for the synthesis of BODIPY is shown in **Figure 5**. The IUPAC numbering system for BODIPY dyes is different to that used for Dipyrromethenes (Dipyrrin) and this can lead to confusion. However, the terms *α-, β*-position and *meso*- are used in just the same way for both system [33]. The IUPAC numbering and conventional nomenclature for BODIPY, Dipyrrin and Dipyrromethane skeleton is shown in **Figure 6**.



**Figure 5**: BODIPY dyes (small, intensely fluorescent systems)



**Figure 6**: The IUPAC numbering and nomenclature for BODIPY, Dipyrrin and Dipyrromethane skeleton

Synthesis of a new BODIPY based fluorogenic probewith an azide connected at the 2-position of the BODIPY ring. Advantages due to intense absorption in visible light, relatively high molar extinction coefficient (ε), biocompatibility, and chemical and photochemical stability encouraged us to explore the chemistry with BODIPY fluorophore [34]. The importance of BODIPY-cholesterol in sterol trafficking in living cells and organisms [35], a substituent consisting of cholesterol side chain was also selected. The procedure is shown in **Scheme 3** [36].



**Scheme 3**: Synthesis of a new boron-Dipyrromethene (BODIPY) based fluorogenic probe

**b. Fluorescent dyes**

BODIPYs are excellent ﬂuorescent dyes and used in various research ﬁelds as labeling reagents, ﬂuorescent switches, chemosensors, light harvesting systems, and dye-sensitized solar cells because of their advantageous photophysical properties such as photostability, high absorption coefficients and high ﬂuorescence quantum yields [37]. BODIPY show application in detection of cysteine and homocysteine in living cells [38] is shown in **Scheme 4**.



**Scheme 4**: Boron-Dipyrromethene show detection of cysteine and homocysteine in living cells

BODIPY derived hydrazones show fluorescent properties [39] in **Figure 7**. Hydrazone formation in BODIPY would also affect the absorption and emission spectra of the BODIPY fluorophore. This aliphatic hydrazone and aromatic hydrazone [40] are shown in **Figure 8**.



**Figure 7**: BODIPY derived hydrazones show fluorescent properties



**Figure 8**: Aliphatic and aromatic hydrazone show fluorescent properties

**c. Photosensitizers**

BODIPY photosensitizers are versatile dyes never tested before in photodynamic application against prokaryotes. Photodynamic therapy requires the presence of photosensitizing agent and harmless light, provided by LED lamps. BODIPY photosensitizers show antimicrobial activity [41, 42]. Synthesis is shown in **Scheme 5**.



**Scheme 5**: BODIPY photosensitizers show antimicrobial activity

**d. Regioselective lithiation of dipyrromethanes**

The feasibility of regioselective lithiation of *N, N’*-dimethyl dipyrromethanes have been synthesized to avoid the formation of anion at more basic nitrogen centers and also show suitable conditions for lithiation of *meso*-position. This is shown in **Scheme 6**.



**Scheme 6**: Regioselective lithiation of *N, N’*-dimethyl dipyrromethane

**e. Development of BODIPY dyes for conjugation with proteins**

BODIPYs are highly valuable compounds in the field of biomolecule labeling due to their chemical stability and photophysical properties. Conjugation of fluorophores to proteins is fundamental to several biotechnological applications and BODIPY dyes started to be used in this field is shown in **Figure 9** [43].



**Figure 9**: Mechanism of amine labeling with succinimidyl ester derivatives

**f. Second-order NLO properties**

To achieve second-order NLO effects, the chromophore contains a conjugated system with strong electron donor and acceptor groups at the opposite ends, creating large dipole in the molecules [44] are shown in **Scheme 7**.



**Scheme 7**: BODIPY show NLO properties

**g. Photophysical properties**

Solvent-dependent photophysical properties of BODIPY have been investigated by means of UV-Vis absorption, steady-state and time-resolved fluorimetry is shown in following compounds (**Scheme 8**) [45].



**Scheme 8**: Solvent-dependent photophysical properties of BODIPY

**Dipyrromethane chemosensors**

Chemosensors are synthetic analogues of [biosensors](https://en.wikipedia.org/wiki/Biosensors), the difference being that biosensors incorporate biological receptors such as antibodies, aptamers or large biopolymers. A molecular sensor or chemosensor is a molecular structure (organic or inorganic complexes) that is used for sensing of an analyzer to produce a detectable change or a [signal](https://en.wikipedia.org/wiki/Signal). The action of a chemosensor, relies on an interaction occurring at the molecular level, usually involves the continuous monitoring of the activity of a chemical species in a given matrix such as solution, air, blood, tissue, waste effluents, drinking water, etc. The application of chemosensors is referred to as chemosensing, which is a form of [molecular recognition](https://en.wikipedia.org/wiki/Molecular_recognition) [46-48]. Chemosensors may also be electrochemically based on small molecules. The development of chemosensors for detecting biologically and environmentally important metal ions, such as Cu2+, Zn2+, Hg2+ and Pb2+ has attracted much attention [49, 50].



**Figure 10**: C-H oxidation and chelation of dipyrromethane by Cu and utilized in the development of rapid colorimetric naked-eye Cu (II) chemosensor

Due to sensitivity concerns, fluorescent chemosensors detecting metal ions using fluorescence enhancement are more easily monitored than those using fluorescence quenching. C-H oxidation based sensor of dipyrromethane and complexation of dipyrrins with metals ions [51].

Charge transfer complexes of dipyrromethanes have also been reported to show excellent selectivity for inorganic anions and neutral molecules [52, 53]. Advancements have been made in the development of sensors for the detection of heavy-transition-metal ions in biological and environmental systems. In particular, the development of probes for heavy-transition-metal selective colorimetric and / or fluorescent sensing systems has become a research hotspot because UV-Vis and fluorescence spectroscopy analyses remain the most commonly used detection methods given their high sensitivity and facile operation. To date, various fluorescent probes based on quinoline, anthracenone, [54, 55] fluorescein, [56, 57] and rhodamine coumarin, fluorophores have been successfully applied to the detection of Zn2+ in vitro and / or in vivo. However, using these sensors to discriminate Zn2+ and Cu2+ [58, 59] remains challenging because Zn2+ are located in the same elemental group and induce similar photophysical changes in sensors. Dipyrromethane is oxidized to dipyrromethene (**Figure 11**). Dipyrromethene can be used as fluorescent “turn-on” Zn sensors. Dipyrromethane shows higher sensitivity to Zn2+ ions [60, 61].



**Figure 11**: Oxidation of dipyrromethane accompanied by coordination with Zn2+

BODIPYs are well established as fluorescence-imaging dyes in diagnostics and share many characteristics with porphyrins and corroles, such as their intense color and fluorescence [62]. Current research is to improve the BODIPY structure towards absorption at higher wavelengths and specifically to increase excited triplet state formation for an application in photo dynamic therapy [63]. This can be done by modifying the BODIPY backbone with halogen atoms or through use of heavy-atom free BODIPY-anthracene dyads.

Dipyrromethanes are of wide interest in organic synthesis and are commonly employed as building blocks for the selective synthesis of *meso*-substituted porphyrinoids as well as *meso*-substituted BODIPYs [64, 65]. Specifically, BODIPYs are easily available from dipyrromethanes via a three-step one-pot synthesis [66, 67]. The stability of *meso*-substituted dipyrromethanes strongly depends on the substitution in the *meso*-position. Electron-withdrawing substituents in this position stabilize the dipyrromethane against decomposition. In addition, electron withdrawing substituents render the dipyrromethane. BODIPY have also been used as accessory pigments in light-harvesting arrays [68].

**Biological applications**

Dipyrromethane compound shows various biological applications are as follows:

**(i) Antibacterial activity and Antioxidant activity**

BODIPY was screened for in vitro antibacterial activity against two gram-negative [*Escherichia coli* (ATCC 25922), *Pseudomonas aeruginosa* (ATCC 27853)] and two gram‒positive [*Staphylococcus aureus* (ATCC 25923) and *Bacillus subtilis* (MTCC 121)] bacterial strains (**Figure 12**) show good antibacterial activity. In biological systems, uncontrolled accumulations of H2O2 leads to the formation of oxygen free radicals which cause immense damage to cells membrane. The antioxidant compounds donate the electrons to H2O2 and neutralize it into H2O molecule [69].



**Figure 12**: BODIPY show antibacterial activity

**(ii) Antimycobacterial activity**

MABA method was used for the assessment of antimycobacterial activity of synthesized compounds against M. *tuberculosis*. All the synthesized compounds show good antimycobacterial activity as shown in **Figure 13** [70].



**Figure 13**: Dipyrromethane show antimycobacterial activity

**(iii) Anti-inflammatory agents**

Dipyrromethanes as structural frame showing promising biological activity as anti-inflammatory agents as shown in **Figure 14**. NO is a small free radical which is produced by nitric oxide syntheses. It is a key signaling molecule that plays an important role in the regulation of many physiological functions, such as host defense, neurotransmission, neurotoxicity, and vasodilation. The physiological or normal generation mediates the tumoricidal and bactericidal actions of macrophages. However, the overproduction of NO can lead to amplification of inflammation, as well as tissue injury. Therefore, inhibition of NO production is a very therapeutic target in the development of anti-inflammatory agent [71].



**Figure 14**: Dipyrromethane show anti-inflammatory activity

**Metal Complex**

A metal complex consists of a central metal atom or ion that is bonded to one or more ligands which are ions or molecules that contain one or more pairs of electrons that can be shared with the metal. A complex ion forms from a metal ion and a ligand because of a Lewis acid-base interaction [72-75]. Reaction of dipyrromethane with divalent metal precursor i.e. MCl2(py)2 in THF solutions to obtained metalated species of dipyrromethane metal complex upon isolation are pale-yellow (Mn), bright orange (Fe), maroon (Co), crimson (Ni), and yellow (Zn) as shown in **Scheme 9**.





**Scheme 9**: Dipyrromethane metal complexes works as fluorescent material

The positively charged metal ion acts as a Lewis acid and the ligand with one or more lone pairs of electrons acts as a Lewis base. Co-ordination compounds contain a central metal atom surrounded by non-metal atoms or groups of atoms called ligands [76-79]. Transition-metal complexes have attracted much attention in recent years because of their potential applications in photocatalysis, photovoltaics, electroluminescence, luminescence bioimaging and molecular sensing, photodynamics, fundamental photochemistry studies, and more recently, triplet-triplet annihilation upconversion [80-82]. The Pt (II) Schiff base complex containing BODIPYs chromophore is shown in **Figure 15** [83].



**Figure 15:** Visible-light-harvesting BODIPYs chromophore

The Nickel diazo-dipyrromethane were synthesized by the reaction of 5, 5’-bisdiazo-dipyrromethane with NiCl2 in methanol in the presence of Et3N [84] is shown in **Scheme 10**.



**Scheme 10**: Synthesis of Nickel diazo-dipyrromethane

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