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Synthesis of Tetravalent Boron Complexes with Pyrazolone-Based Ligand and their Drug-Likeness

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Abstract: ***Boron chemistry is one of the most interesting fields for synthetic organic chemists. In present investigation we synthesized 3-methyl-1-phenyl-2-pyrazolone from the reaction of ethyl acetoacetate and phenyl hydrazine. 3-methyl-1-phenyl-2-pyrazolone treated with benzene diazonium chloride to furnish arylazo pyrazolone ligand. Boric acid reacts with isopropanol in benzene to form boron isopropoxide as a colourless viscous liquid. Refluxing of boron isopropoxide with catechol in benzene gives isopropoxy-benzodioxaboron. isopropoxy-benzodioxaboron and 3-methyl-1-phenyl-2-pyrazolone was refluxed in benzene to give four coordinated boron complexes. The structures of all synthesized compounds were confirmed by physical and analytical data.***

All synthesized arylazo pyrazolone ligand were evaluated for drug-likeness under Lipinski's, Ghose, Veber, Egan and Muegge's rules and may have good drug candidature.

Keyword: Boron Complexes, Pyrazolone, Drug-likeness

I. INTRODUCTION

Boron is an important element that plays a key role in many biological processes [1]. The chemistry of tetra-coordinated Boron complexes is the field of interest for chemists.

Boron has an empty p-orbital and is electrophilic in nature. Boron can coordinat with nucleophiles, which enables it to interact with hydroxyl and amino groups present in biological systems such as enzyme amino acid residues, nucleic acids, and carbohydrates [2]. Boron compounds also showed the various biological properties as antibacterial [3], antifungal [4], antitumor [5], antioxidant [6] enzyme inhibitor [7-8] etc.

In present scenario, boron complexes have become one of the most popular fields of researcher due to the interesting coordination and other properties [9-10]. Various boron complexes with different properties were prepared using variety of ligands, such as bidentate thiazole-bridged 1,5-bidentate nitrogen ligands [11], amino phenolic N,O-ligands [12]; tridentate salicylidenehydrazinopyridine ligands [13], dianilidopyridine pincer ligand [14], bis(2-hydroxybenzyl)methylamine ligands [15], shiff-base [16]; and tetradentate N_2O_2-type dipyrrin ligands [17].

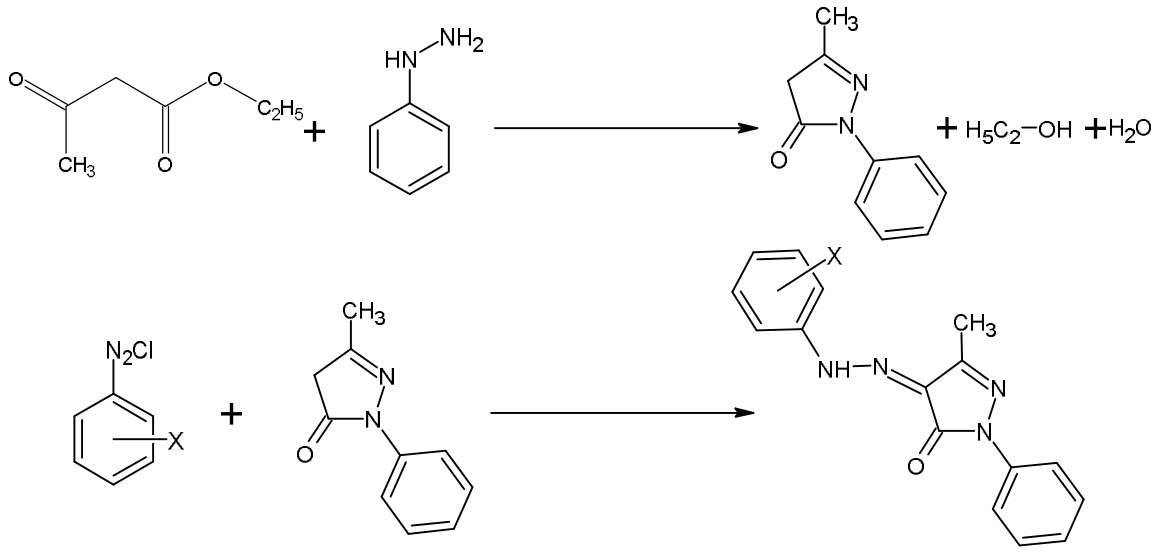
The use of chelating ligands has played an important role in the design, synthesis, and application of functional boron complexes. Because of the potential advantages of Lewis acidity, unique electronic and enhanced flexibility, boron complexes are widely applied in fields such as catalysis [18].

II. EXPERIMENTAL

A. Synthesis of 5-methyl-2-phenyl-4-(2-phenylhydrazinylidene)-2,4-dihydro-3H-pyrazol-3-one(HPAP-1).-

5-methyl-2-phenyl-4-(2-phenylhydrazinylidene)-2,4-dihydro-3*H*-pyrazol-3-one was prepared by heating freshly distilled ethyl acetoacetate (1 mole) and phenyl hydrazine (0.8 mole) in presence of catalytic amount of acetic acid. The crude mehylphenyl pyrazolone obtained was filtered, washed with ether and recrystalized by 50% aqueous alcohol.

Phenyl diazonium chloride were prepared by diazotization of the respective phenyl amine in concentrate HCl and $NaNO_2$ at $0\text{-}5^0$ C. the diazonium solution of the respective amine was added to a acetone-water solution(1:1) of 3-Methyl-1-phenyl-5-pyrazolone in presence of sodium acetate as buffer. The temperature during the addition was maintained between $5\text{-}10^0$ C. the crude 5-methyl-2-phenyl-4-(2-phenylhydrazinylidene)-2,4-dihydro-3H-pyrazol-3-one obtained was filtered and recrystallized with ethanol.

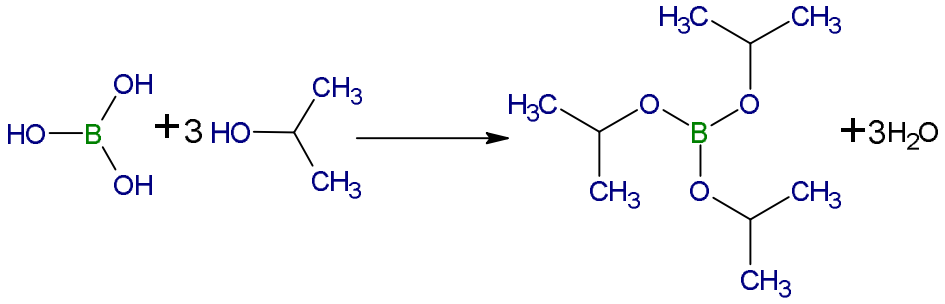


Spectral data of synthesized compounds-

1. 5-methyl-2-phenyl-4-(2-phenylhydrazinylidene)-2,4-dihydro-3*H*-pyrazol-3-one(HPAP-1).
IR in KBR:- ν N-H 3420, ν C=O 1660, ν C=N 1595, ν N-N 1550.
^{1}H-NMR δ 2.25 s(3H, CH_3), δ 6.9-8 m (Ar-H), δ 13.8 s(1H, NH).
2. 5-methyl-4-[2-(2-methylphenyl)hydrazinylidene]-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one(HPAP-2).
IR in KBR:- ν N-H 3425, ν C=O 1655, ν C=N 1590, ν N-N 1545.
^{1}H-NMR δ 2.27 s(3H, CH_3), δ 7-8 m (Ar-H), δ 13.7 s(1H, NH).
3. 5-methyl-4-[2-(4-methylphenyl)hydrazinylidene]-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one(HPAP-3).
IR in KBR:- ν N-H 3415, ν C=O 1660, ν C=N 1595, ν N-N 1545.
^{1}H-NMR δ 2.24 s(3H, CH_3), δ 6.8-7.9 m (Ar-H), δ 13.8 s(1H, NH).
4. 4-[2-(2-chlorophenyl)hydrazinylidene]-5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one(HPAP-4).
IR in KBR:- ν N-H 3420, ν C=O 1665, ν C=N 1600, ν N-N 1550.
^{1}H-NMR δ 2.26 s(3H, CH_3), δ 7-8 m (Ar-H), δ 13.6 s(1H, NH).
5. 4-[2-(4-chlorophenyl)hydrazinylidene]-5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one(HPAP-5).
IR in KBR:- ν N-H 3415, ν C=O 1660, ν C=N 1605, ν N-N 1555
^{1}H-NMR δ 2.26 s(3H, CH_3), δ 6.9-8 m (Ar-H), δ 13.7 s(1H, NH)
6. 4-[2-(4-bromophenyl)hydrazinylidene]-5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one(HPAP-6).
IR in KBR:- ν N-H 3420, ν C=O 1655, ν C=N 1605, ν N-N 1550
^{1}H-NMR δ 2.28 s(3H, CH_3), δ 7.1-8 m (Ar-H), δ 13.8 s(1H, NH)

B. Synthesis of Boron isopropoxide

Boron isopropoxide was prepared by the reaction of boric acid with isopropanol in benzene. The water formed in the reaction was removed azeotropically. After completion of the reaction the excess solvent was initially removed by distillation under reduced pressure. The product obtained was distilled at 125-135º/760 mm Hg to get boron isopropoxide as a colourless viscous liquid.



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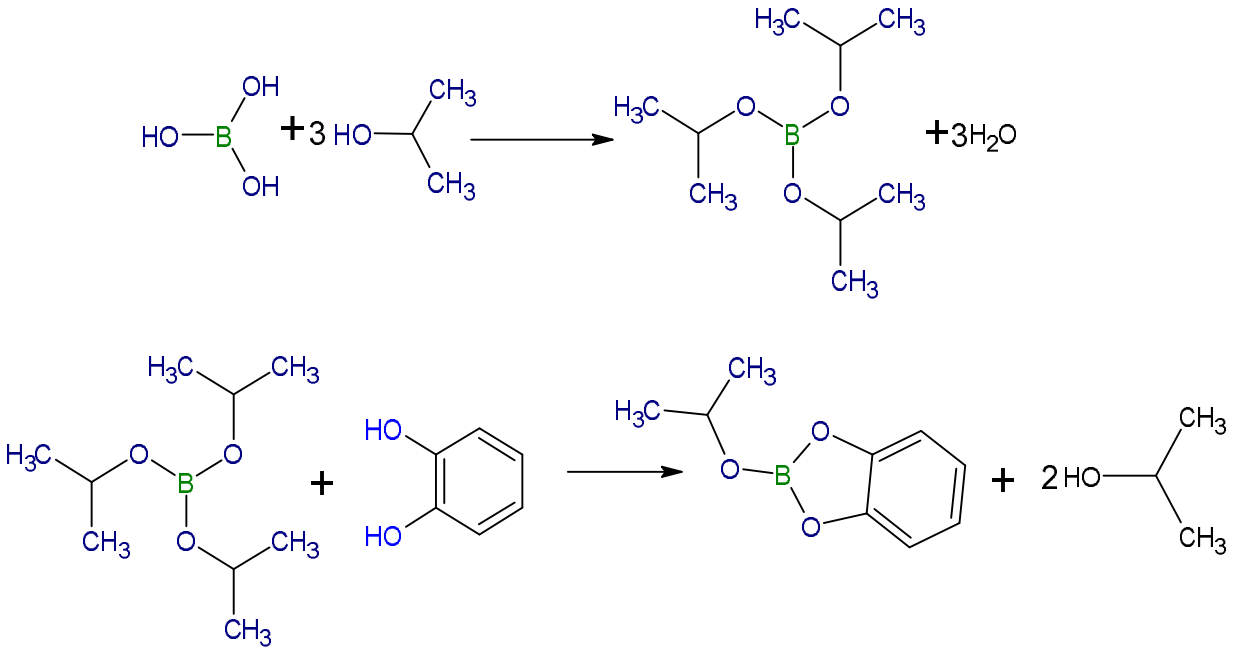
C. Synthesis of 2-Isopropoxy-1,3,2-benzodioxaborole:-

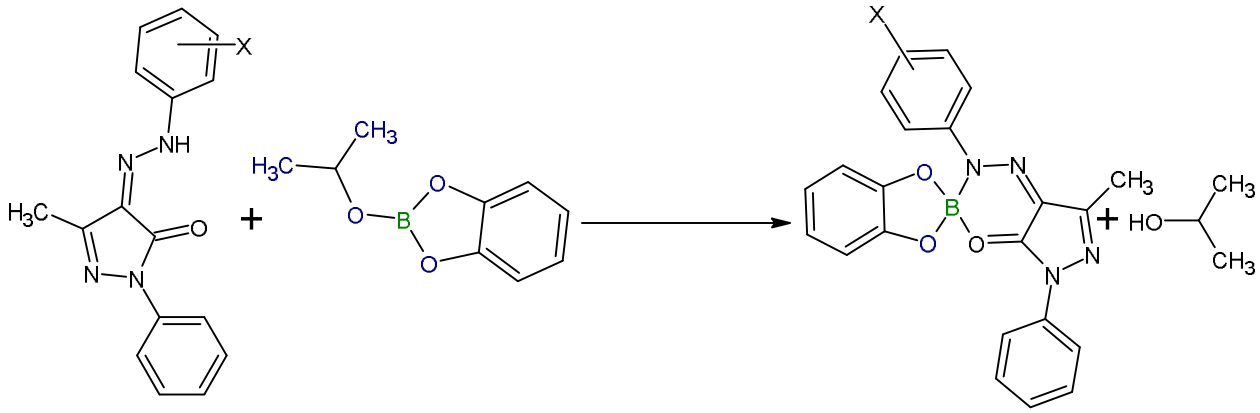
To a solution of boron isopropoxide (0.1 mole) in dry benzene, catechol (0.1 mole) was added. The reaction mixture was refluxed for about 5 hours. The progress of the reaction was followed by estimating the liberated isopropanol azeotropically fractioned with benzene. After completion of the reaction, excess solvent was removed under reduced pressure and 2-isopropoxy-1,3,2-benzodioxaborole was obtained by distillation under reduced pressure.



1) Synthesis of 2-(3-methyl-1-phenyl-4-arylazo-5-pyrazolonate)-1,3,2-benzodioxaborale complexes-

The alkoxy precursor 2-Isopropoxy-1,3,2-benzodioxaborate undergoes facile reaction with 5-methyl-2-phenyl-4-(2-phenylhydrazinylidene)-2,4-dihydro-3H-pyrazol-3-one derivatives in benzene to give 2-(3-methyl-1-phenyl-4-arylazo-5-pyrazolonate)-1,3,2-benzodioxaborate.

The isopropanol librated in the reaction was fractionated out azeotropically with benzene. The progress and completion of the reaction was followed by estimating the liberated isopropanol iodometrically.



2) Spectral data of complexes-

1. 2-Isopropoxy-1,3,2-benzodioxaborate and 5-methyl-2-phenyl-4-(2-phenylhydrazinylidene)-2,4-dihydro-3*H*-pyrazol-3-one complex

IR in KBr:- ν C=N 1620, ν C---O and C---N 1565, ν B-O 1285

^{1}H-NMR:- δ 2.24 s(3H, CH_3), δ 6.79-8.12 m (Ar-H),

^{11}B-NMR:- δ 7.38

2. 2-Isopropoxy-1,3,2-benzodioxaborate and 5-methyl-4-[2-(2-methylphenyl) hydrazinylidene]-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one complex
IR in KBr:- ν C=N 1615, ν C---O and C---N 1560, ν B-O 1280.
^{1}H-NMR:- δ 2.23 s(3H, CH_3), δ 6.74-8.07 m (Ar-H), δ 2.33 s (3H-CH_3)
3. 2-Isopropoxy-1,3,2-benzodioxaborate and 5-methyl-4-[2-(4-methylphenyl) hydrazinylidene]-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one complex
IR in KBr:- ν C=N 1620, ν C---O and C---N 1560, ν B-O 1280
^{1}H-NMR:- δ 2.22 s(3H, CH_3), δ 6.75-8.07 m (Ar-H), δ 2.34 s (3H-CH_3)
4. 2-Isopropoxy-1,3,2-benzodioxaborate and4-[2-(2-chlorophenyl)hydra zinylidene]-5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one complex
IR in KBr:- ν C=N 1620, ν C---O and C---N 1565, ν B-O 1285
^{1}H-NMR:- δ 2.23 s(3H, CH_3), δ 6.69-8.06 m (Ar-H),
5. 2-Isopropoxy-1,3,2-benzodioxaborate and 4-[2-(4-chlorophenyl)hydra zinylidene]-5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one complex
IR in KBr:- ν C=N 1615, ν C---O and C---N 1560, ν B-O 1280
^{1}H-NMR:- δ 2.23 s(3H, CH_3), δ 6.79-8.08 m (Ar-H),
^{11}B-NMR:- δ 7.23
6. 2-Isopropoxy-1,3,2-benzodioxaborate and 4-[2-(4-bromophenyl)hydra zinylidene]-5-methyl-2-phenyl-2,4-dihydro-3*H*-pyrazol-3-one complex
IR in KBr:- ν C=N 1620, ν C---O and C---N 1560, ν B-O 1280
^{1}H-NMR:- δ 2.22 s(3H, CH_3), δ 6.75-8.086 m (Ar-H).

3) Physicochemical Properties and drug drug-likeness

The physicochemical properties and drug-likeness of pyrazoline ligand MPAP-1 to MPAP-6 were in-silico predicted by the SwissADME website.

III. RESULT AND DISCUSSION

Reaction of $B(OPr^i)_3$ with catechol in 1:1 molar ratio in benzene yields 2-Isopropoxy-1,3,2-benzodioxaborale. The alkoxy precursor 2-Isopropoxy-1,3,2-benzodioxaborale react with 3-Methyl-1-phenyl-4-aryl azo-2-pyrazoline-5-one to furnished 2-(3-methyl-1-phenyl-4-arylazo-5-pyrazolonate)-1,3,2-benzodioxaborale complex. The ability of the 2-Isopropoxy-1,3,2-benzodioxaborale precursor to react with 3-Methyl-1-phenyl-4-aryl azo-2-pyrazoline-5-one and formed complex can be attributed to the electronic effects of the coordinate dianions. The catecholate dianion is expected to exhibit greater electron withdrawing effect due to which 2-Isopropoxy-1,3,2-benzodioxaborale is shows higher Lewis acidity and reactivity towards 3-Methyl-1-phenyl-4-aryl azo-2-pyrazoline-5-one to form tetravalent boron complexes. IR spectral data shows medium intense peak at 715-745 cm^{-1} and 1280-1300 cm^{-1} for ν(B←O) and ν(B−O) stretching mode respectively. The 1H-NMR spectra of complexes shows the absence of the N-H proton signals in the spectra of the free azo ligand. The analytical and spectral data of the complexes in the present investigation give evidence for tetra-coordinated complexes with N, O bonding of the azo ligand.

A. Physical Data of Complexes

S No.	Reactants		% Yield	Pr^i-OH Found	% Elemental Analysis Found(Calc.)				M.P. °C
	(-OC_6H_4O-)$BOPr^i$ (gms, mmol)	HPAP Arylazo (gms, mmol)			% C	% H	% N	% B	
1	1.17, 6.61	C_6H_5NN: 1.83, 6.61	87	0.28	66.42 (66.65)	4.28 (4.32)	14.08 (14.13)	2.65 (2.72)	194
2	1.29, 7.28	o-$CH_3C_6H_4NN$: 2.12, 7.28	82	0.38	67.25 (67.30)	4.59 (4.66)	13.58 (13.64)	2.57 (2.63)	173
3	1.47, 8.30	p-$CH_3C_6H_4NN$: 2.42, 8.30	84	0.37	67.25 (67.30)	4.59 (4.66)	13.58 (13.64)	2.57 (2.63)	180

4	1.35, 7.62	o-ClC_6H_4NN: 2.37, 7.62	85	0.39	61.24 (61.32)	3.68 (3.74)	12.92 (13.00)	2.45 (2.50)	167
5	1.18, 6.66	p-ClC_6H_4NN: 2.07, 6.66	82	0.31	61.24 (61.32)	3.68 (3.74)	12.92 (13.00)	2.45 (2.50)	174
6	1.37, 7.74	p-BrC_6H_4NN: 2.76, 7.74	87	0.38	55.45 (55.58)	3.31 (3.39)	11.69 (11.78)	2.19 (2.27)	177

B. Physicochemical Properties and drug drug-likeness

The physicochemical properties express the description of the structure of molecules like molecular weight, molecular refractivity, number of rotatable bond, heavy atoms, topological polar surface area, hydrogen bond acceptors and donor.

The bioavailability radar includes the following six physicochemical properties:

(1) Lipophilicity (XLOGP3 between −0.7 and +5.0).

(2) Size (molecular weight between 150 and 500 g/mol).

(3) Polarity (the total polar surface area between 20 and 130 Å2).

(4) Solubility (log S not higher than 6).

(5) Saturation (fraction Csp3 not less than 0.25).

(6) Flexibility (the number of rotatable bonds not more than 9).

Properties		**HPAP**-1	**HPAP**-2	**HPAP**-3	**HPAP**-4	**HPAP**-5	**HPAP**-6
M.Weight		278.31	292.34	292.34	312.75	312.75	357.20
Heavy atoms		21	22	22	22	22	22
Arom. Heavy atoms		12	12	12	12	12	12
Fraction Csp3		0.06	0.12	0.12	0.06	0.06	0.06
Rotatable bond		3	3	3	3	3	3
H- Bond acceptors		3	3	3	3	3	3
H- Bond Donor		1	1	1	1	1	1
Molar Refractivity		90.81	95.77	95.77	95.82	95.82	98.51
TPSA Å^2		57.06	57.06	57.06	57.06	57.06	57.06
Lipophilicity	MlogP	2.30	2.54	2.54	2.81	2.81	2.93
	WlogP	1.92	2.23	2.23	2.58	2.58	2.69
	XlogP3	3.25	3.61	3.61	3.87	3.87	3.93

Drug likeness of compounds established based on the physicochemical properties to find oral drug candidates (Daina *et al*., 2017) [19]. There are five different rule-based filters which are defined as follows:

1) Lipinski's rule includes molecular weight ≤ 500, lipophilicity ≤ 4.15, hydrogen bond acceptors ≤ 10 and hydrogen bond donors ≤ 5 (Lipinski *et al*., 2001) [20].

2) Ghose's rule includes molecular weight in between 160 to 480, WLOGP (lipophilicity) in between−0.4 to 5.6, molar refractivity in between 40 to 130, and number of atoms in between 20 to 70 (Ghose *et al*., 1999) [21].

3) Veber's rule includes the number of rotatable bonds are less than 10 and the total polar surface area is less than 140 (Veber *et al*., 2002) [22].

4) Egan's rule includes WLOGP (Lipophilicity) less than 5.88 and the total polar surface area less than 131.6 (Egan *et al*., 2000) [23].

5) Muegge's rule includes molecular weight in between 200 to 600, XLOGP3 (lipophilicity) in between −2 to 5, the total polar surface area less than 150, the number of rings less than 7, the number of carbon greater than 4, the number of heteroatoms greater than 1, the number of rotatable bonds are less than 15, the hydrogen bond acceptors less than 10, and the hydrogen bond donors less than 5 (Muegge *et al*., 2001) [24]

Rule	MPAP-1	MPAP-2	MPAP-3	MPAP-4	MPAP-5	MPAP-6
Lipinski	Yes	Yes	Yes	Yes	Yes	Yes
Ghose	Yes	Yes	Yes	Yes	Yes	Yes
Veber	Yes	Yes	Yes	Yes	Yes	Yes
Egan	Yes	Yes	Yes	Yes	Yes	Yes
Muegge	Yes	Yes	Yes	Yes	Yes	Yes

The result of drug-likeness evaluation based on various physicochemical properties included rules of pyrazolines (HPAP-1 to HPAP-6) is depicted in Table 3, and on the basis of result we can conclude that:

All the pyrazolines (HPAP-1 to HPAP-6) are in agreement with the Lipinski's, Ghose, Veber, Egan and Muegge's rules and that may have good drug candidature.

IV. CONCLUSION

The present study reports the successful synthesis of a series of tetra-coordinated Boron complexes derived from the reaction of 2-isopropoxy-1,3,2-benzodioxaborole with 5-methyl-2-phenyl-4-(2-phenylhydrazinylidene)-2,4-dihydro-3H-pyrazol-3-one ligands. The formation of these complexes is facilitated by the strong electron-withdrawing nature of the catecholate dianion, which enhances the Lewis acidity of the boron center and promotes coordination with the azo-pyrazolone ligand.

Furthermore, the evaluation of physicochemical properties revealed that the synthesized compounds possess molecular weights, lipophilicity, hydrogen bonding capacity, and polar surface area within the acceptable limits for drug-like molecules. According to Lipinski's rule of five and other drug-likeness filters, the compounds exhibit favourable characteristics for potential oral bioavailability.

Overall, the study demonstrates that the synthesized boron complexes show promising structural and physicochemical properties, suggesting their potential as candidates for further investigation in medicinal and pharmaceutical applications.

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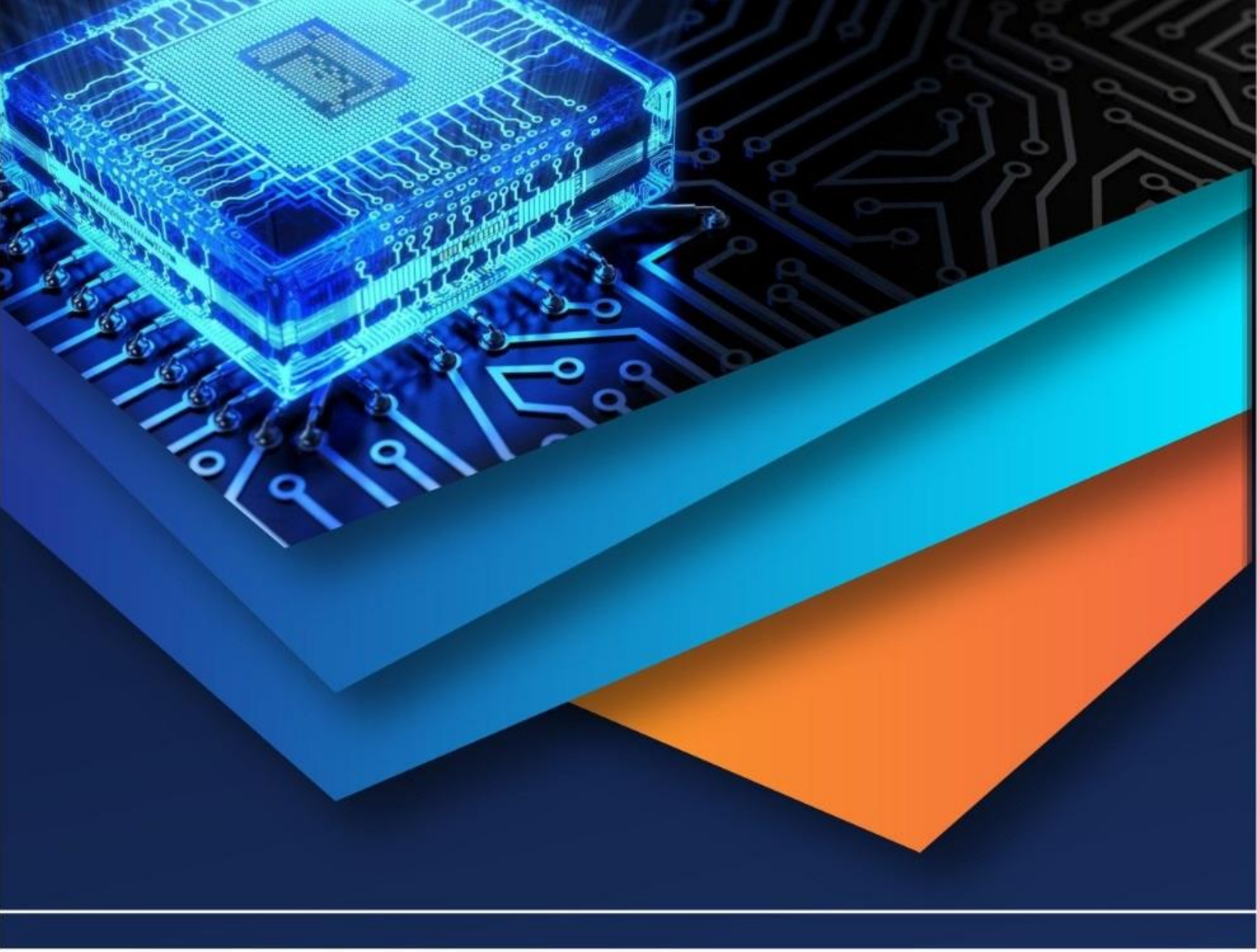

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