



THREE COMPONENT SYNTHESIS OF 4,4(ARYLMETHYLENE) BIS (1H-PYRAZOL- 5-OLS)

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Abstract:

Pyrazoles have attracted particular attention due to the diverse biological activities associated with this heterocyclic system, and some have been shown to be cytotoxic to several human cell lines. Several drugs currently on the market have this heterocycle as the key structural motif, and some have been approved for the treatment of different types of cancer.

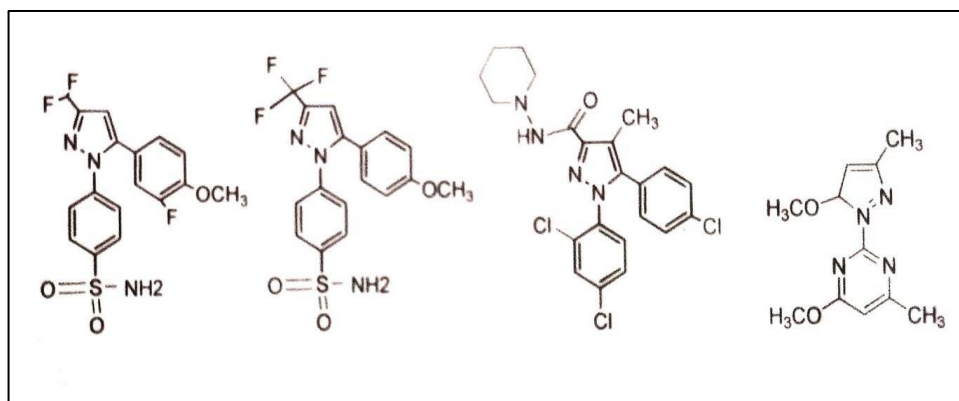
Keywords: 4,4(arylmethylen) bis (1H-pyrazol-5- ols),anitioxidant,pharmaceutical,anti-inflammatory,

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Introduction:

Among widespread heterocyclic compounds, nitrogen heterocycles occupy a diverse position because of their widespread natural abundance and extensive biological as well as pharmaceutical importance. Pyrazole derivatives, one of the widely used bioactive scaffolds among the heterocycles and studied in the development of insecticides, fungicides, herbicides, dyes, and reagents. Particularly some of the leading commercial

drugs (1-4) based on the pyrazole scaffold includes celecoxib, lonazolac and rimonabant[1]. The term pyrazole was first coined by Ludwig Knorr in 1883. Due to its composition and unique pharmacological effects on human beings, they are classified as alkaloids. 1- Pyrazolyl-alanine was the first natural pyrazole isolated from watermelon seeds in the year 1959 [2].

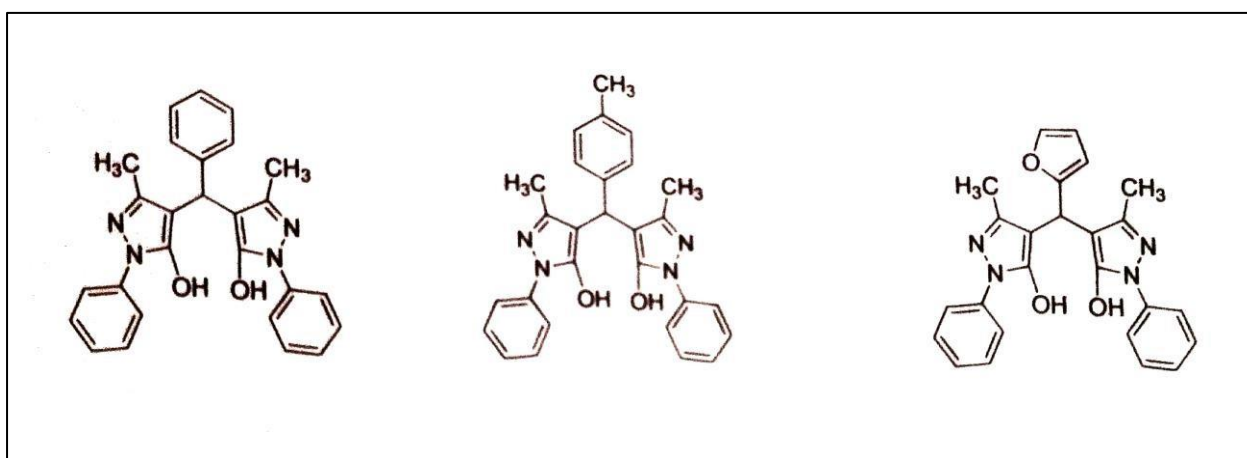


Bis(heterocyclyl)methanes represent an important class of compounds that constitute the building blocks of natural and synthetic porphyrins [3]. These occur widely in various natural products and showed versatile biological and pharmacological activities [4]. Among

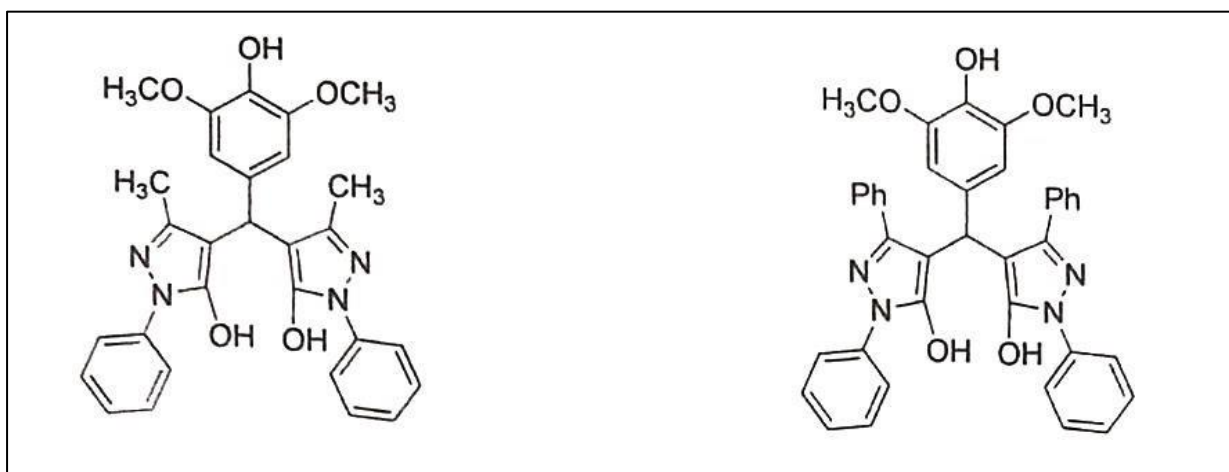
them 4,4'- (arylmethylene)bis(3-methyl-1- phenyl-1H-pyrazol-5-ol)s have a wide spectrum of approved pharmaceutical activity and display agrochemical properties [5].

Review of literature:- Biological Significance :
1. Antiviral activity:

Kuppusamy Sujatha et al. reported efficient and eco-friendly method for the synthesis of 4,4'-(arylmethylene)bis(1H-pyrazol-5-ols) by using CAN in water. All synthesized compound were evaluated for in vitro antiviral activity against peste des petits ruminant virus (PPRV). Among all synthesized compound (5) and (6) shows good antiviral activity and compound (7) showed excellent antiviral activity and found to be more potent than standard ribavirin [5].


2. Antioxidant activity

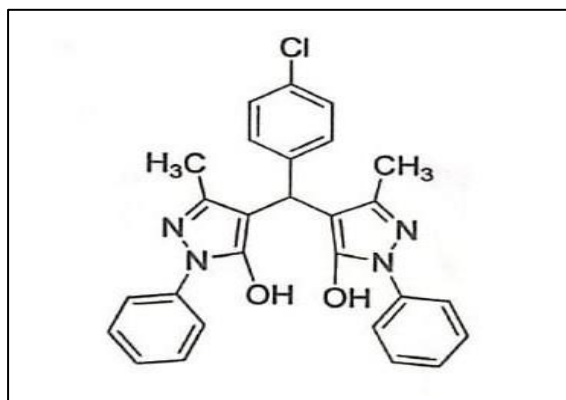
Yang Xiaohui et al. described synthesis and antioxidant activity of 4,4'-(arylmethylene)bis(1H-pyrazol-5-ols). The synthesized compound were screened for in vitro antioxidant activities by N,N-diphenyl-N'-picrylhydrazyl (DPPH) and 2,2'-azino bis(3-ethylthiazoline-sulphonic acid) diammonium salt (ABTS+) radical scavenging assays. Almost all compounds showed good DPPH and ABTS+ radical scavenging activities. Among all compounds (8) and (9) showed the better antioxidant activities against DPPH and ABTS+[6].





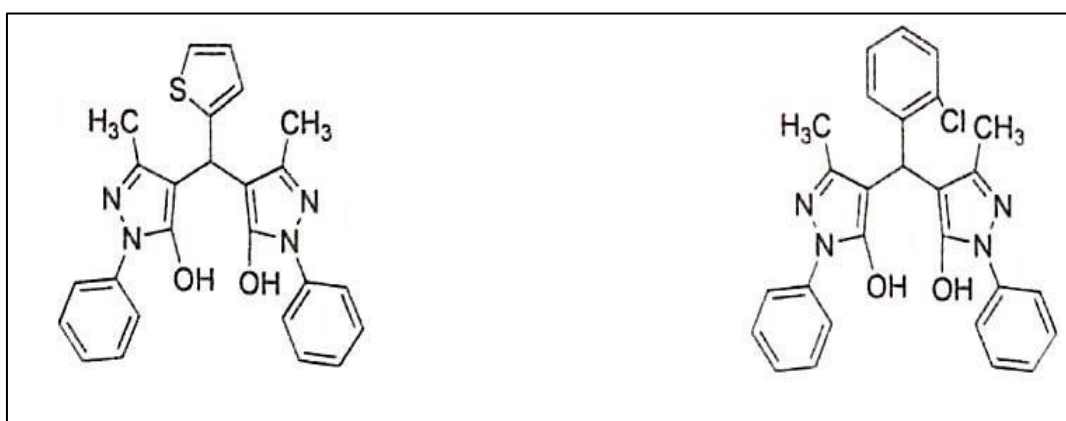
3. Metal chelating and extracting agent:

M.Abbasi-Tarighat et al. reported D simple and sensitive spectrophotometric method to the simultaneous determination of Mn ²⁺ and Fe³⁺ in foods, vegetable and water sample with the help of artificial neural networks (ANNs). It depended on high sensitive bispyrazole (10) based ligand shown below [7].



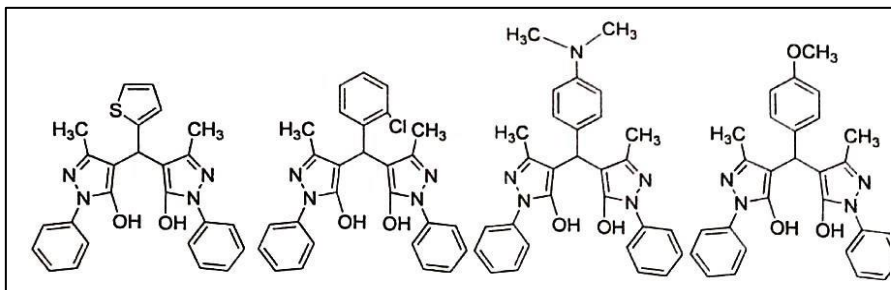
4. Anti-inflammatory activity:

Pravin S. Mahajan and coworkers reported synthesis of 4,4'(arylmethylene)bis(1Hpyrazol-5-ols) by using L-proline through pseudo multicomponent reaction. All the synthesized compounds were screened for anti-inflammatory activity with comparison of standard drug Diclofenac sodium. Compound (11) showed potent anti-inflammatory activities with EC₅₀ of 10.87 ± 0.65 whereas, compound (12) showed good activity with EC₅₀ of 12.25 ± 2.03 µg/mL compared to DFS [8].



5. Antimicrobial activity:

Pravin S. Mahajan and coworkers reported antimicrobial activity in addition to antiinflammatory activity. All synthesized compound evaluated for in vitro antimicrobial activity against gram- negative bacterial strains Escherichia coli, and Micrococcus luteus, Pseudomonas Aeruginosa and Staphylococcus aureus and antifungal strain Aspergillus niger and Candida albicans compared to the used standard. It was found that compound (13, 14, 15 and 16) showed good anti-inflammatory activity [8]



4,4'-(Arylmethylene)bis(1H-pyrazol-5-ols) are applied as fungicides, pesticides and dyestuffs. The condensation of aldehydes with two equivalents of 3-methyl-1-phenyl-5-pyrazolone is a conventional chemical approach to 4,4'-(arylmethylene)bis(1H-pyrazol-5-ols). Many catalysts have been used for this transformation such as xanthan sulfuric acid, phosphomolybdic acid, silica sulfuric acid, 3-aminopropylated silica gel, sodium dodecyl sulfate, [Cu(3,4-tmtppa)](MeSO₄), PEG-SO₃H⁺, cellulose sulfuric acid⁺, lithium hydroxide monohydrate, 1,3,5-tris(hydrogensulfato)benzene⁺, sulfuric acid ([3-(3-silicapropyl)sulfanyl]propyl)ester⁺, ethylenediammonium diacetate, [Sipmim]HSO₄, 1, TEBA⁺, ceric ammonium nitrate, silica-bonded S-sulfonic acid, 2-hydroxyethylammonium acetate, 1,3-disulfonic acid imidazolium tetrachloroaluminate and 1-sulfonypyridinium chloride⁺. Catalyst-free protocol and the electrocatalytic procedure were also applied to the preparation of 4,4'-(arylmethylene)bis(1H-pyrazol-5-ols)-2. All of the aforementioned procedures include two main steps: (1) 3-methyl-1-phenyl-5-pyrazolone should be synthesized from phenylhydrazine and ethyl acetoacetate firstly, and (2) then 3-methyl-1-phenyl-5-pyrazolone reacted with aldehydes. Even though 4,4'-(arylmethylene)bis(1H-pyrazol-5-ols) could be synthesized by these methods, most of the methods suffer from limitations such as long reaction time, use of expensive catalysts, the requirement of special apparatus, tedious work-up procedures and noncompliance with green chemistry protocols. Therefore, finding an efficient and eco-friendly protocol for the preparation of 4,4'-(arylmethylene)bis(1H-pyrazol-5-ols) is of obvious importance.

In green chemistry, elimination of volatile organic solvents in organic synthesis is a most important goal. Solvent-free conditions makes synthesis simpler, save energy, and prevent solvent waste, hazards, and toxicity²⁸⁻²⁹

In continuation of our work on the development of useful synthetic methodologies³⁷⁻³⁸, we report herein, an alternative protocol for the one-pot three-component synthesis of 4,4'-(arylmethylene)bis(1H-pyrazol-5-ols) derivatives starting directly from aldehydes, phenylhydrazine/hydrazine hydrate and ethyl acetoacetate using acetic acid as an efficient, cost-effective and catalyst under in ethanol (Scheme 1).

Scheme 1. Acetic acid catalyzed synthesis of 4,4'-(arylmethylene)bis(1H-pyrazol-5-ols)

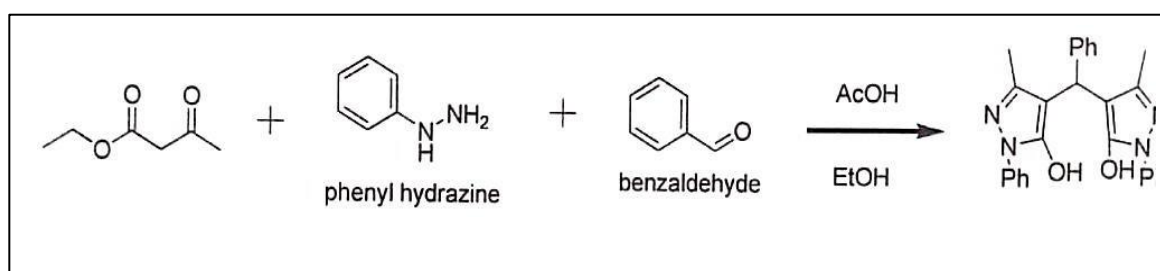
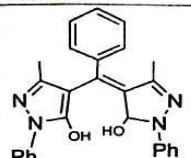
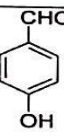
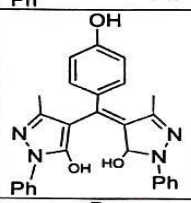
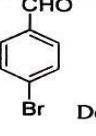
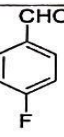
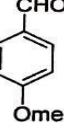
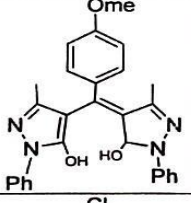
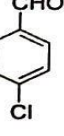
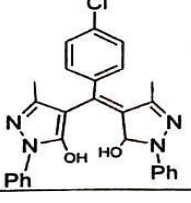
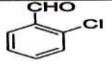
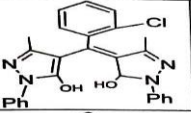
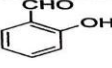
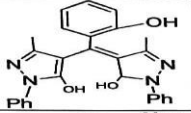
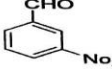
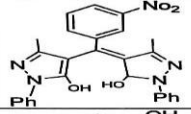
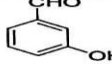
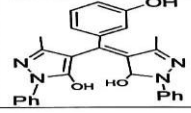




Table :

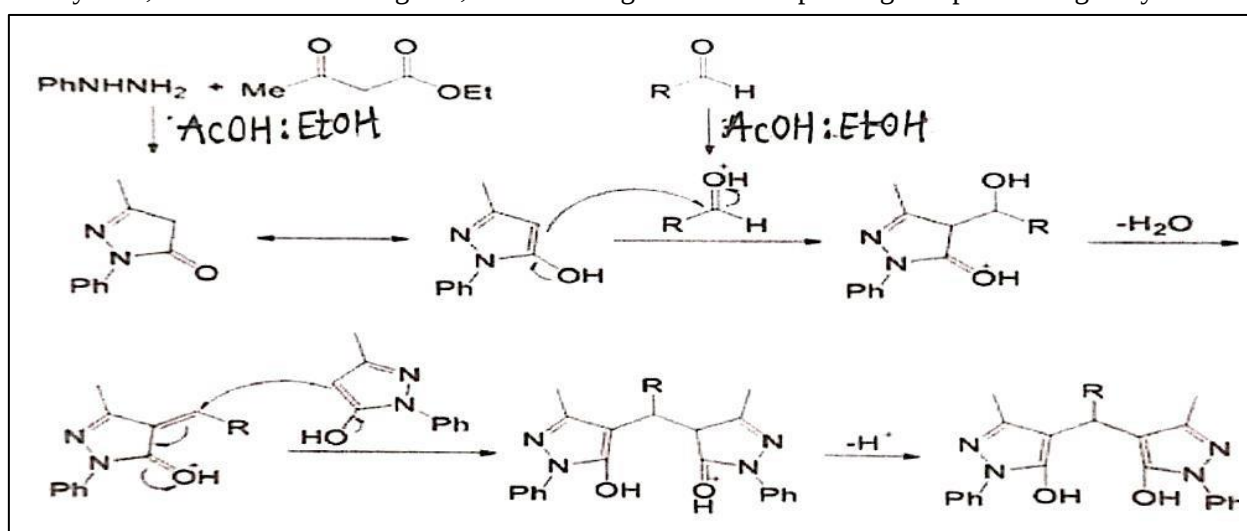
Sr.No	Derivative	Product	Time	Yield
1			60 min	72 %
2			75 min	80 %
3			65 min	75 %
4			70 min	70 %
5			75 min	65 %
6			60 min	85 %

7			65 min	68 %
8			80 min	82 %
9			70 min	80 %
10			64 min	85 %

Results and Discussion:

In order to optimize the reaction conditions, the reaction of phenylhydrazine, ethyl acetoacetate and 2,4-dichlorobenzaldehyde was selected as a model reaction. The reaction was performed in different solvents. With 10 percent acetic acid in ethanol at reflux temperature optimum yields of the products were obtained.

In order to establish the generality, the catalyst was successfully applied to the reaction by using different aromatic aldehydes with a wide range of ortho-, meta and para-substitutions under the optimized reaction conditions. The results are summarized in Table 1. It is clear from this table that, high product yields were obtained with aromatic aldehydes containing electron- donating and electron-withdrawing substituents. Furthermore, the reaction is compatible in the presence of various functional groups such as Cl, -OCH₃, -NO₂ and -OH. When changing phenylhydrazine into hydrazine hydrate, a similar result was given; the reaction gave the corresponding compounds in good yields.



Scheme 2. Plausible mechanism for the synthesis of 4,4'-(arylmethylene) bis(1H-pyrazol-5-ols) catalyzed by Acetic acid.

Conclusion:

In summary, this paper describes a convenient and efficient process for the synthesis of 4,4'-(arylmethylene)bis(1H-pyrazol-5-ols) through the three-component coupling of aldehydes, ethyl acetoacetate and phenylhydrazine/hydrazine hydrate using [acetic acid as catalyst. High yields of products, short reaction time, easily available and cheap catalyst, simple experimental and isolation procedures, eco-friendly reaction conditions make this methodology a valid contribution to the existing processes in the field of 4,4'-(arylmethylene)bis(1H-pyrazol-5-ols) derivatives synthesis.

Experimental:

Material and instruments:

General procedure for the synthesis of 4,4'-(arylmethylene)bis(1H-pyrazol-5-ols)

A mixture of aldehyde (5 mmol), ethyl acetoacetate (10 mmol), phenylhydrazine (10 mmol), and acetic acid() in ethanol was stirred at reflux conditions. The progress of the reaction was monitored by TLC using EtOAc/petroleum ether (1/2) as eluent. During the reaction process, a solid product spontaneously formed. After completion of the reaction, the reaction mixture was cooled to room temperature. The resulting solid was recrystallized by using ethanol (95%) to afford the

pure product. All the products are known and were identified by comparison of their physical and spectroscopic data with those of authentic samples. The spectral data of products are given below:

4,4'-(Phenylmethylene)bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2, entry 1): ¹H NMR (DMSO-d, 400 MHz) 8: 2.32 (s, 6H, 2CH₃), 4.96 (s, 1H, CH), 7.17-7.30 (m, 7H, ArH), 7.44 (t, J=7.6 Hz, 4H, ArH), 7.71 (d, J=8.0 Hz, 4H, ArH). IR (KBr) v: 3358, 3061, 2965, 2582, 1596, 1495, 1414, 1272, 1074, 792, 734, 689 cm⁻¹.

4,4'-[(4-Methylphenyl)methylene]-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2, entry 2) ¹H NMR (DMSO-d, 400 MHz) & 2.24 (s, 4H, CH₃), 2.30 (s, 6H, 2CH₃), 4.90 (s, 1H, CH), 7.09 (d, J=8.4 Hz, 2H, ArH), 7.13 (d, J=8.0 Hz, 2H, ArH), 7.24 (J=7.2 Hz, 2H, ArH), 7.44 (t, J=7.6 Hz, 4H, ArH), 7.70 (d, J=7.6-12.4 Hz, 4H, ArH). IR (KBr) v: 3413, 3051, 2928, 1604, 1581, 1507, 1414, 1299, 806, 752, 695 cm⁻¹.

4,4'-[(4-Methoxyphenyl)methylene]-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2, entry 3): ¹H NMR (DMSO-d, 400 MHz) : 2.32 (s, 6H, 2CH₃), 3.72 (s, 3H, CH₃O), 4.93 (s, 1H, CH), 6.87 (d, J=8.8 Hz, 2H, ArH), 7.18 (d, J=8.4 Hz, 2H, ArH), 7.23-7.31 (m, 2H, ArH), 7.45-7.49 (m, 4H, ArH), 7.72-7.73 (m, 4H, ArH), IR (KBr) v: 3437, 3068, 2918, 2559, 1606, 1584, 1510, 1411, 1378, 1039, 801, 755 cm⁻¹.

4,4'-[(4-Fluorophenyl)methylene]-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2, entry 4): ¹H NMR (DMSO-d, 400 MHz) 8: 2.32 (s, 6H, 2CH₃), 4.96 (s, 1H, CH), 7.09 (t, J=8.0 Hz, 2H, ArH), 7.24-7.32 (m, 4H, ArH), 7.45 (t, J=7.6 Hz, 4H, ArH), 7.72 (d, J=8.0 Hz, 4H, ArH). IR (KBr) v: 3445, 3080, 288, 1590, 1495, 1401, 1380, 1308, 1121, 902, 842, 788, 690 cm⁻¹.

4,4'-[(3-Methoxyphenyl)methylene]-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2, entry 5): ¹H NMR

(DMSO-d, 400 MHz) 6: 2.42 (s, 6H, 2CH₃), 3.51 (s, 3H, CH₃O), 4.71 (s, 1H, CH), 6.55 (d, J=8.8 Hz, 2H, ArH), 6.66-6.72 (m, 2H, ArH), 7.89 (m, 1H, ArH), 7.32-7.45 (m, 4H, ArH), 7.73 (m, 4H, ArH). IR (KBr) v: 3428, 3085, 2920, 1580, 1508, 1410, 1133, 1025, 804, 685 cm⁻¹.

4,4'-[(3-Nitrophenyl)methylene]-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2, entry 6): ¹H NMR (DMSO-d, 400 MHz) 8: 2.30 (s, 6H, 2CH₃), 4.95 (s, 1H, CH), 7.2 (t, J=7.2 Hz, 2H, ArH), 7.37 (t, J=8.0 Hz, 2H, ArH), 7.44 (t, J=8.8 Hz, 2H, ArH), 7.67 (d, J=8.0 Hz, 4H, ArH), 8.07 (d, J=6.4 Hz, 4H, ArH). IR (KBr) v: 3078, 2918, 1599, 1523, 1502, 1346, 1269, 1093, 758, 734, 696 cm⁻¹.

4,4'-[4-Hydroxy-3-methoxyphenyl)methylene]-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2, entry 7): ¹H NMR (DMSO-d, 400 MHz) 5: 2.33 (s, 6H, 2CH₃), 3.69 (s, 3H, CH₃O), 4.87 (s, 1H, CH), 6.71-6.74 (m, 2H, ArH), 6.88-6.89 (m, 1H, ArH), 7.24-7.26 (m, 2H, ArH), 7.43-7.47 (m, 4H, ArH), 7.72-7.74 (m, 4H, ArH), 8.81 (s, 1H, OH). IR (KBr) v: 3213, 3071, 2561, 1609, 1507, 1422, 1264, 1131, 1044, 816, 789, 760, 693 cm⁻¹.

4,4'-[(2-Chlorophenyl)methylene]-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2, entry 8): ¹H NMR (DMSO-d, 400 MHz) 8: 2.29 (s, 6H, 2CH₃), 5.14 (s, 1H, CH), 7.22-7.33 (m, 4H, ArH), 7.41 (d, J=8.0 Hz, 1H, ArH), 7.46 (t, J=7.6 Hz, 4H, ArH), 7.70 (d, J=8.0 Hz, 4H, ArH), 7.80 (d, J=7.2 Hz, 1H, ArH). IR (KBr) v: 3435, 3066, 2916, 1615, 1562, 1503, 1404, 1374, 1310, 839, 752, 695 cm⁻¹.

4,4'-[(4-Chlorophenyl)methylene]-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2, entry 9): ¹H NMR (DMSO-d, 400 MHz) 8: 2.30 (s, 6H, 2CH₃), 4.98 (s, 1H, CH), 7.22-7.28 (m, 4H, ArH), 7.35 (d, J=8.4 Hz, 2H, ArH), 7.44 (t, J=8.0 Hz, 4H, ArH), 7.71

(d, J-8.0 Hz, 4H, ArH). IR (KBr) ν : 3432, 3068, 2924, 1601, 1496, 1412, 1296, 1196, 1094, 1019, 835, 752, 691 cm^{-2} .

4,4'-[(4-Bromophenyl)methylene]bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2. entry 10): ^1H NMR (DMSO- d_6 400 MHz) δ : 2.32 (s, 6H, 2CH), 4.95 (s, 1H, CH), 7.19-7.27 (m, 4H, ArH), 7.42-7.48 (m, 6H, ArH), 7.70 (d, J-8.0 Hz, 4H, ArH). IR (KBr) ν : 3422, 3066, 2922, 2546, 1598, 1484, 1407, 1293, 1013, 809, 747, 687 cm^{-2}

4,4'-[(2,4-Dichlorophenyl)methylene]bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2, entry 11): ^1H NMR (DMSO- d_6 400 MHz) δ : 2.28 (s, 6H, 2CH₃), 5.09 (s, 1H, CH), 7.25 (t, J-7.2 Hz, 2H, ArH), 7.40-7.46 (m, 5H, ArH), 7.56 (d, J-2.0 Hz, 1H, ArH), 7.69 (d, J-8.0 Hz, 4H, ArH), 7.75 (d, J-8.4 Hz, 1H, ArH). IR (KBr) ν : 3425, 3060, 2919, 1595, 1573, 1498, 1471, 1380, 1295, 1105, 843, 754, 690 cm^{-1} .

4,4'-[(4-Nitrophenyl)methylene]-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2. entry 12): ^1H NMR (DMSO- d_6 400 MHz) δ : 2.28 (s, 6H, 2CH), 5.06 (s, 1H, CH), 7.18 (t, 2H, J-7.2 Hz, ArH), 7.38 (LJ=7.2 Hz, 4H, ArH), 7.45 (d, J-8.4 Hz, 2H, ArH), 7.64 (d, J-8.0 Hz, 4H, ArH), 8.10 (d, J-8.8 Hz, 2H, ArH). IR (KBr) ν : 3423, 3071, 2925, 1599, 1518, 1502, 1348, 1299, 835, 759, 693 cm^{-1} .

4,4'-[(2-Hydroxyphenyl)methylene]-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2. entry 13): ^1H NMR (DMSO- d_6 400 MHz) δ : 2.28 (s, 6H, 2CH), 5.16 (s, 1H, CH), 6.70-6.74 (m, 2H, ArH), 6.96 (t, J-7.2 Hz, 1H, ArH), 7.22 (L, J=7.2 Hz, 2H, ArH), 7.42 (t, J=8.0 Hz, 4H, ArH), 7.57 (d, J-7.2 Hz, 1H, ArH), 7.71 (d, J-7.8 Hz, 4H, ArH). IR (KBr) ν : 3427, 3066, 2928, 2832, 1603, 1578, 1501, 1454, 1372, 1231, 754, 690 cm^{-1} .

4,4'-[(4-Hydroxyphenyl)methylene]bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2. entry 14): ^1H NMR

(DMSO- d_6 400 MHz) δ : 2.30 (s, 6H, 2CH), 4.84 (s, 1H, CH), 6.66 (d, J-8.8 Hz, 2H, ArH),

7.04 (d, J-8.8 Hz, 2H, ArH), 7.22-7.26 (L, J-7.2 Hz, 2H, ArH), 7.44 (t, J-8.0 Hz, 4H, ArH), 7.71 (d, J-8.0 Hz, 4H, ArH), 9.16 (s, 1H, OH). IR (KBr) ν : 3413, 3158, 2969, 1597, 1502, 1427, 1275,

819, 756, 692 cm^{-1}

4,4'-[(2-Methoxyphenyl)methylene]-bis(3-methyl-1-phenyl-1H-pyrazol-5-ol) (Table 2, entry 15):

^1H NMR (DMSO- d_6 400 MHz) δ : 2.26 (s, 6H, 2CH), 3.79 (s, 3H, OCH₃), 5.18 (s, 1H, CH), 6.82-6.93 (m, 2H, ArH), 7.11-7.24 (m, 3H, ArH), 7.41 (1, J-7.2 Hz, 4H, ArH), 7.60 (d, J-7.6 Hz, 1H, ArH), 7.67-7.70 (m, 4H, ArH). IR (KBr) ν : 3427, 3062, 2922, 1598, 1575, 1497, 1241, 756 cm^{-1} .

4,4'-(Phenylmethylene)bis(3-methyl-1H-pyrazol-5-ol) (Table 2, entry 16): ^1H NMR (DMSO- d_6 400 MHz) δ : 2.07 (s, 6H, 2CH), 4.82 (s, 1H, CH), 7.09-7.14 (m, 3H, ArH), 7.19-7.22 (m, 2H, ArH). IR (KBr) ν : 3296, 2971, 1612, 1522, 1494, 1380, 1049, 825, 778, 717 cm^{-1}

4,4'-[(3-Nitrophenyl)methylene]bis(3-methyl-1H-pyrazol-5-ol) (Table 2. entry 17): ^1H NMR (DMSO- d_6 400 MHz) δ : 2.11 (s, 6H, CH), 4.99 (s, 1H, CH), 7.54-7.57 (m, 2H, ArH), 7.96 (s, 1H, ArH), 8.03 (s, 1H, ArH). IR (KBr): 3419, 2961, 1599, 1447, 1390, 1182, 837, 795, 764 cm^{-1} .

4,4'-[(4-Chlorophenyl)methylene]bis(3-methyl-1H-pyrazol-5-ol) (Table 2. entry 18): ^1H NMR (DMSO- d_6 400 MHz) δ : 2.08 (s, 6H, CH), 4.82 (s, 1H, CH), 7.12 (s, 2H, ArH), 7.27 (m, 2H, ArH). IR (KBr): 3437, 1614, 1488, 1388, 1094, 757 cm^{-1}

4,4'-[(4-Hydroxyphenyl)methylene]bis(3-methyl-1H-pyrazol-5-ol) (Table 2. entry 19): ^1H NMR (DMSO- d_6 400 MHz) δ : 2.06 (s, 6H, CH), 4.71 (s, 1H, CH), 6.58 (d, J-8.4 Hz, 2H, ArH), 6.90 (d, J-8.4 Hz, 2H, ArH), 9.04 (s, 1H, OH). IR (KBr): 3266, 1561, 1514, 1466, 1400, 1174, 872, 786, 731 cm^{-1} .

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