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Ionic Liquid Mediated Synthesis And Anticancer Activity Of 5-((1,3-Diphenyl-1h-Pyrazol-4 Yl)Methylene) Dihydroindenones

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Abstract
A series of 5-((1,3-diphenyl-1H-pyrazol-4-yl)methylene)dihydroindenone derivatives were synthesised by the Claisen-Schmidt condensation of 1,3-diphenyl-1H-pyrazole-4-carbaldehyde with 2,3-dihydro-1H-inden-1-one using Ionic liquid under both conventional heating method and microwave irradiation method. All the synthesised derivatives were screened for their anticancer activity against three different human cell lines as cervix (SiHa), breast (MDA-MB-231) and pancreatic carcinoma (PANC-1) using the Sulforhodamine B assay method.

Keywords: *Ionic liquid, microwave irradiation, 2,3-dihydro-1H-inden-1-one, pyrazole, and anticancer activity*

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I. Introduction:

Environmental concerns in synthetic chemistry have led to a reconsideration of reaction methodologies. One obvious route to reduce waste entails generation of chemicals from reagents in the absence of solvents. Therefore, the design of green processes with no use of hazardous and expensive solvents, Synthetic organic chemists have given particular emphasis to "solvent-free" reactions. As a result, numerous novel reactions are discovered to proceed effectively and cleanly in the solid state or in the absence of solvents. Solvent-free reactions have become more and more popular recently, mostly due to decreased chemical pollution, lower costs, and simpler processes. Although this method of chemical synthesis seems straightforward, it has several drawbacks, the most significant of which being the importance of diffusion and reactant interactions. Furthermore, it is never certain if reactions occurring in the solid form will result in the same compounds as those produced when solvents are present. One of the most prized structures in medicinal chemistry, indanone is frequently linked to a variety of pharmacologically effective molecules. Indanones have proven to have a wide range of biological action. Additionally, they are excellent synthons for the synthesis of numerous heterocyclic and carbocyclic compounds that are used as synthetic intermediates for a variety of pharmaceuticals and natural products[1-3]. Anti-inflammatory[4], analgesic[5], antimicrobial[6], anticholinergic[7], dopaminergic[8], anticancer[9], and antimalarial[10] actions are just a few of their significant biological effects. There are many bioactive natural compounds that include the indanone moiety. Marine cyanobacterium and Pteris ensiformisburm were used to isolate key natural compounds including 1-methoxy-6-methyl-3-oxo2,3-dihydro-1H-indene-4-carbaldehyde (I) and Pterosin B (II)[11,12]. Paucifloral F (III), a possible antiviral polyphenolic derivative, and its isomer isopaucifloral F, a potential anti-osteoporosis agent, are two further significant natural products with the indanone ring system[13,14]. Researchers have created a library of pharmacologically active indanones during the past few years as a result of the indanone core's significance in biology. The novel indonone analogs have just recently been described as antiviral and antibacterial agents[15,16]. Pyrazole is an important biologically active scaffold that possesses nearly all types of biological activities[17]. Pyrazoles are a promising scaffold for many anticancer agents[18]. Several clinical anticancer therapeutics, such as crizotinib, ruxolitinib, niraparib, encorafenib, and darolutamide, currently consist of a pyrazole moiety[19]. Therefore, in the past decades, many pyrazolyl analogues were synthesized and tested as anticancer agents. On the other hand, Ionic liquids (ILs) are developed as a sustainable alternative to most of the volatile organic solvents. As the research on ILs progress, they emerged also as suitable catalysts and reagents. ILs possess unique set of properties with respect to their cationic and anionic components, which allows ILs to be used in varied fields. They overcome many of the traditional limitations related to conventional organic synthesis. Encouraged by the above different applications, we have synthesised a series of 5-((1,3-diphenyl-1H-pyrazol-4-yl)methylene)dihydroindenone derivatives by the Claisen-Schmidt condensation of 1,3-diphenyl-1H-pyrazole-4-carbaldehyde with 2,3-dihydro-1*H*-inden-1-one using Ionic liquid under both conventional heating method and microwave irradiation method, reported reaction time, and obtained yield data and then evaluated the resulting molecules for their anticancer potential.

II. Result and discussion:

A series of 5-((1,3-diphenyl-1H-pyrazol-4-yl)methylene)dihydroindenones were synthesized in three chemical transformation such as synthesis of 1-(1-Arylethylidene)-2-phenylhydrazines, 1-Phenyl-3-(pyridin-3-yl)-1H-pyrazole-4-carbaldehyde and 5-((1,3-diphenyl-1H-pyrazol-4-yl)methylene)dihydroindenone. 1-(1-Arylethylidene)-2-phenylhydrazines **(3a-3i)** were synthesized by the condensation of aryl methyl ketones **(1a-1i)** with phenyl hydrazine **(2)** in the presence of acetic acid in the methanol medium at heating condition to get solid 1-(1-Arylethylidene)-2-phenylhydrazines **(3a-3i).** 3-Aryl-1-phenyl-1H-pyrazole-4-carbaldehydes **(4a-4i)** were synthesized by reacting 1-(1-Arylethylidene)-2-phenylhydrazines **(3a-3i)** with Vilsmeir-Haack reagent at room temperature for 8hr in good yields. Finally the compounds 5-((1,3-diphenyl-1H-pyrazol-4-yl)methylene)dihydroindenones **(6a-6i)** by the condensation of 3-Aryl-1-phenyl-1H-pyrazole-4-carbaldehyde with 2,3-dihydro-1*H*-inden-1-one in the presence of N-(4-sulfonic acid) butyl triethylammonium hydrogen sulphate ([**TEBSA][HSO$_4$**]) under both conventional heating and microwave irradiation method to get corresponding 5-((1,3-diphenyl-1H-pyrazol-4-yl)methylene)dihydroindenones **(6a-6i)**. Scope and generality of the cascade reaction, the indanone reacted with pyrazole aldehydes under the optimized reaction conditions to afford desired products (**6a-6i)** in good yield. ^{1}HNMR, ^{13}CNMR, and mass spectrum data analyses were used to describe the newly synthesised compounds. The ^{1}HNMR spectra of compound 6a showed two distinctive peaks at 4.12 ppm integrated for two protons allocated to the methylene group and a singlet peak at 8.98 ppm integrated for one proton attributed to the pyrazole ring proton. The compound 6a revealed two distinctive peaks in the ^{13}C NMR spectra at 31.8 and 192.5, which were ascribed to methylene and cyclic carbonyl carbon, respectively, and the spectrum indicated the requisite number of carbon peaks. The compound 6a mass spectrum revealed a peak at 363 m/z values as $[M+H]^+$.

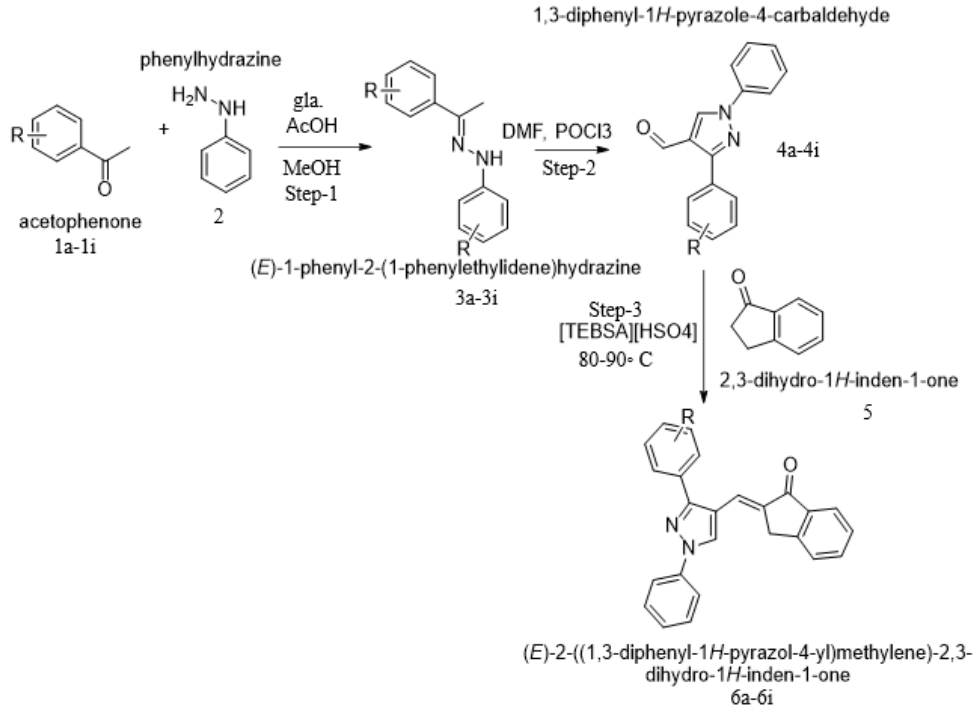

Table-1: Physical data of 5-((1,3-diphenyl-1H-pyrazol-4-yl)methylene)dihydroindenones **(6a-6i)**

Entry	Ar	M.P. (°C)	Reaction time		Yield(%)	
			Conven (hr)	MWI (min)	Conven	MWI
6a	Phenyl	141-143	6	8	75	82
6b	4-Fluorophenyl	135-137	6	8	72	83
6c	4-Chlorophenyl	139-141	6	8	76	82
6d	4-Brorophenyl	150-152	6	8	74	80
6e	2,4-dichlorophenyl	145-147	6	8	76	82
6f	4-Methylphenyl	133-135	6	8	71	82
6g	4-Methoxyphenyl	131-133	6	8	72	80
6h	4-Nitrophenyl	147-149	6	8	70	78
6i	4-Hydroxyphenyl	167-169	6	8	70	78

ANTICANCER ACTIVITY:

Di-aryl-pyrazolyl-dihydroindenone derivatives **(6a-6i)** were evaluated to determine their anticancer activity against three different human tumor cell lines as cervix (SiHa), breast (MDA-MB-231) and pancreatic carcinoma (PANC-1) (SiHa, MDA-MB-231 and PANC-1 using the Sulforhodamine B assay method. Doxorubicin is used as standard. The GI_{50} values are listed in **Table-2**. From the screening results, among the compounds **6a**, **6f** and **6g** showed potent activity against as Cervix (SiHa) cell lines with compared to doxorubicin, the compounds **6b**, **6f** and **6h** showed good anticancer activity on breast (MDA-MB-231) cell lines and the compounds **6e** and **6i** exhibited maximum anticancer activity on pancreatic carcinoma (PANC-1).

Table-2: Anticancer activity of di-aryl-pyrazolyl-dihydroindenone derivatives (GI_{50} values).

Compound	SiHa	MDA-MB-231	PANC-1
6a	1.52	1.81	3.96
6b	--	1.52	3.96
6c	--	2.05	--
6d	2.01	--	3.98
6e	2.31	--	2.77
6f	1.92	1.21	4.71
6g	1.85	2.23	3.56
6h	--	1.36	4.11
6i	--	2.67	2.89
Doxorubicin	2.31	1.15	3.10

Experimental: Melting points were determined in open capillary tubes and are uncorrected. The purity of the compounds was checked by TLC using precoated silica gel plates 60_{254}(Merck). IR (KBr) spectra were recorded on a Shimadzu FT-IR-8400s spectrophotometer. ^{1}H& ^{13}C NMR spectrums were recorded on Bruker Avance II 400 MHz spectrometer using tetramethylsilane as an internal standard. Mass spectra were recorded on a GCMS-QP 1000 EX mass spectrometer.

General procedure for the preparation of 5-((1,3-diphenyl-1H-pyrazol-4-yl)methylene)dihydroindenones (6a-6i):

Microwave irradiation method: A mixture of 3-Aryl-1-phenyl-1H-pyrazole-4-carbaldehydes **(4a-4i)** (1.0mmol) and 2,3-dihydro-1*H*-inden-1-one **(5)** (1.0 mmol) in [TEBSA][HSO_4] (0.1mmol) was taken in a microwave vial and irradiated at 120W for 8 min. Reaction progress monitored by TLC. After completion of the reaction. Cooled to RT and added ice cold water to get solid to give corresponding 5-((1,3-diphenyl-1H-pyrazol-4-yl)methylene)dihydroindenones **(6a-6i)**

Conventional stirring method: A mixture of 3-Aryl-1-phenyl-1H-pyrazole-4-carbaldehydes **(4a-4i)** (1.0 mmol), 2,3-dihydro-1*H*-inden-1-one **(5)** (1.0 mmol) and [TEBSA][HSO_4] (0.1 mmol) in ethanol (10.0 ml) was taken in a RB flask and stirred at 80-90°C for up to 6 hours. Reaction progress monitored by TLC. After completion of the reaction. Cooled to RT poured into ice cold water to get to get solid to give corresponding 5-((1,3-diphenyl-1H-pyrazol-4-yl)methylene)dihydroindenones **(6a-6i).**

Spectral data:

(E)-2-((1,3-diphenyl-1H-pyrazol-4-yl)methylene)-2,3-dihydro-1H-inden-1-one (6a):

M.P.: 141-143 °C;

IR spectrum, *v*, cm^{-1}: 1219, 1593, 1668 and 2920;

^{1}H NMR spectrum, δ, ppm: 4.12 s (2H, CH_2), 7.40–7.44 t (1H, ArH), 7.46–7.51 m (3H, ArH), 7.53–7.60 m (4H, ArH), 7.66–7.74 m (4H, ArH), 7.75–7.77 d (1H, ArH), 8.05-8.07 d (2H, ArH), 8.98 s (1H, ArH).

^{13}C NMR spectrum, δ_C, ppm: 31.8, 116.6, 119.1, 122.2, 123.5, 126.3, 127.2, 127.5, 129.0, 129.5, 130.5, 131.0, 131.8, 134.2, 134.7, 137.7, 138.8, 149.3, 152.6, 192.5.

Mass spectrum: *m/z* 363 $[M + H]^+$.

(E)-2-((3-(4-Fluorophenyl)-1-phenyl-1H-pyrazol-4-yl)methylene)-2,35-dihydro-1H-inden-1-one (6b):
M.P.: 135-137 °C;
IR spectrum, *v*, cm^{-1}: 1222, 1595, 1666 and 2922;
^1H NMR spectrum, δ, ppm: 4.11 s (2H, CH_2), 7.27–7.29 d (2H, ArH), 7.42–7.45 m (1H, ArH), 7.65–7.61 m (4H, ArH), 7.69–7.75 m (3H, ArH), 7.76–7.80 m (2H, ArH), 8.04–8.06 d (2H, ArH), 8.96 s (1H, ArH). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 114.6, 118.9, 121.5, 123.4, 125.9, 126.9, 127.5, 128.6, 128.9, 129.3, 131.7, 133.8, 134.5, 137.5, 138.5, 149.2, 153.9, 192.4. Mass spectrum: *m/z* 381 $[M + H]^+$.

(E)-2-((3-(4-Chlorophenyl)-1-phenyl-1H-pyrazol-4-yl)methylene)-2,3-dihydro-1H-inden-1-one (6c):
M.P.: 139-141 °C;
IR spectrum, *v*, cm^{-1}: 1218, 1594, 1669 and 2925;
^1H NMR spectrum, δ, ppm: 4.11 s (2H, CH_2), 7.40–7.47 m (3H, ArH), 7.66–7.63 m (2H, ArH), 7.65–7.72 m (6H, ArH), 7.75–7.77 d (1H, ArH), 8.04–8.05 d (2H, ArH), 8.97 s (1H, ArH). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 116.5, 119.0, 122.5, 123.3, 126.5, 126.9, 127.4, 128.8, 129.0, 129.3, 129.7, 131.5, 134.2, 134.8, 137.5, 138.7, 149.2, 153.5, 192.4. Mass spectrum: *m/z* 397 $[M + H]^+$.

(E)-2-((3-(4-Bromophenyl)-1-phenyl-1H-pyrazol-4-yl)methylene)-2,3-dihydro-1H-inden-1-one (6d):
M.P.: 150-152°C;
IR spectrum, *v*, cm^{-1}: 1222, 1592, 1669 and 2927;
^1H NMR spectrum, δ, ppm: 4.11 s (2H, CH_2), 7.42–7.47 m (3H, ArH), 7.66–7.62 m (3H, ArH), 7.67–7.71 m (3H, ArH), 7.75–7.78 m (3H, ArH), 8.03–8.05 d (2H, ArH), 8.97 s (1H, ArH). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 116.6, 119.0, 122.6, 123.4, 126.4, 127.1, 127.5, 128.7, 128.8, 128.9, 129.5, 131.8, 134.0, 134.7, 137.8, 138.9, 149.4, 153.9, 192.6. Mass spectrum: *m/z* 441 $[M + H]^+$.

(E)-2-((3-(2,4-dichlorophenyl)-1-phenyl-1H-pyrazol-4-yl)methylene)-2,3-dihydro-1H-inden-1-one (6e):
M.P.: 145-147 °C;
IR spectrum, *v*, cm^{-1}: 1226, 1595, 1667 and 2925;
^1H NMR spectrum, δ, ppm: 4.11 s (2H, CH_2), 7.38–7.45 m (2H, ArH), 7.47–7.50 m (1H, ArH), 7.61–7.66 m (3H, ArH), 7.65–7.71 m (4H, ArH), 7.74–7.76 d (1H, ArH), 8.04–8.05 d (2H, ArH), 8.96 s (1H, ArH). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 116.3, 118.8, 121.5, 123.3, 125.2, 126.5, 1296.9, 127.3, 128.6, 129.5, 130.2, 130.5, 133.4, 134.6, 136.7, 137.4, 139.1, 149.5, 153.2, 192.4. Mass spectrum: *m/z* 432 $[M + H]^+$.

(E)-2-((3-(4-Methylphenyl)-1-phenyl-1H-pyrazol-4-yl)methylene)-2,3-dihydro-1H-inden-1-one (6f):
M.P.: 133-135 °C;
IR spectrum, *v*, cm^{-1}: 1221, 1592, 1666 and 2923;
^1H NMR spectrum, δ, ppm: 2.45 s (3H, OCH_3), 4.11 s (2H, CH_2), 7.25–7.27 d (2H, ArH), 7.41–7.51 m (3H, ArH), 7.56–7.61 m (3H, ArH), 7.66–7.78 m (4H, ArH), 8.04–8.06 d (2H, ArH), 8.96 s (1H, ArH). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 24.5, 115.9, 121.3, 123.4, 125.9, 127.1, 127.4, 128.9, 129.4, 130.2, 130.9, 132.0, 133.9, 134.5, 137.5, 138.6, 149.2, 152.5, 192.4. Mass spectrum: *m/z* 377 $[M + H]^+$.

(E)-2-((3-(3-Methoxyphenyl)-1-phenyl-1H-pyrazol-4-yl)methylene)-2,3-dihydro-1H-inden-1-one (6g):
M.P.: 136-138 °C;
IR spectrum, *v*, cm^{-1}: 1221, 1596, 1668 and 2920;
^1H NMR spectrum, δ, ppm: 3.84 s (3H, OCH_3), 4.11 s (2H, CH_2), 7.10–7.12 d (1H, ArH), 7.27–7.29 d (1H, ArH), 7.42–7.49 m (3H, ArH), 7.56–7.62 m (4H, ArH), 7.67–7.75 m (3H, ArH), 8.04–8.06 d (2H, ArH), 8.96 s (1H, ArH). ^{13}C NMR spectrum, δ_C, ppm: 31.7, 111.7, 113.2, 116.5, 119.1, 123.1, 126.6, 127.5, 128.5, 129.1, 129.5, 130.0, 131.3, 133.9, 137.5, 138.5, 149.5, 152.4, 153.6, 192.5. Mass spectrum: *m/z* 393 $[M + H]^+$.

(E)-2-((3-(4-Methoxyphenyl)-1-phenyl-1H-pyrazol-4-yl)methylene)-2,3-dihydro-1H-inden-1-one (6h):
M.P.: 131-133 °C;
IR spectrum, *v*, cm^{-1}: 1219, 1595, 1662 and 2920;
^1H NMR spectrum, δ, ppm: 3.85 s (3H, OCH_3), 4.13 s (2H, CH_2), 7.13–7.15 d (2H, ArH), 7.39–7.50 m (3H, ArH), 7.57–7.60 m (3H, ArH), 7.67–7.78 m (4H, ArH), 8.04–8.06 d (2H, ArH), 8.97 s (1H, ArH). ^{13}C NMR

spectrum, δ_C, ppm: 31.8, 55.6, 108.5, 116.5, 118.8, 122.4, 123.1, 125.9, 126.8, 127.3, 128.4, 128.9, 129.5, 131.3, 133.9, 134.5, 136.8, 149.1, 152.5, 153.5, 192.3. Mass spectrum: *m/z* 393 $[M + H]^+$.

(E)-2-((3-(4-Nitrophenyl)-1-phenyl-1H-pyrazol-4-yl)methylene)-2,3-dihydro-1H-inden-1-one (6i):
M.P.: 147-149 °C;
IR spectrum, *v*, cm^{-1}: 1223, 1595, 1668 and 2923;
^1H NMR spectrum, δ, ppm: 4.17 s (2H, CH_2), 7.42–7.47 m (3H, ArH), 7.61–7.90 m (5H, ArH), 8.08–8.10 d (2H, ArH), 8.13–8.15 m (3H, ArH), 8.37–8.38 d (2H, ArH), 9.06 s (1H, ArH). ^{13}C NMR spectrum, δ_C, ppm: 31.8, 117.1, 119.2, 122.5, 123.3, 125.9, 126.9, 127.4, 127.9, 129.0, 129.4, 129.9, 130.2, 130.6, 136.9, 138.9, 142.3, 145.3, 149.5, 152.3, 192.4. Mass spectrum: *m/z* 408 $[M + H]^+$.

III. Conclusion:

We have successfully developed a new green synthetic ionic liquid based synthesis of 5-((1,3-diphenyl-1H-pyrazol-4-yl)methylene)dihydroindenone from 3-Aryl-1-phenyl-1H-pyrazole-4-carbaldehydes and 2,3-dihydro-1*H*-inden-1-one under both conventional and microwave irradiation method. The microwave irradiation method to be an easy, gave higher yield with lower reaction times, greater selectivity and eco-friendlier. Screened anticancer activity all the synthesised derivatives, among the compounds **6a**, **6f** and **6g** showed potent activity against as Cervix (SiHa) cell lines with compared to doxorubicin, the compounds **6b**, **6f** and **6h** showed good anticancer activity on breast (MDA-MB-231) cell lines and the compounds **6e** and **6i** exhibited maximum anticancer activity on pancreatic carcinoma (PANC-1).

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