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Research Article

MICROWAVE-ASSISTED SYNTHESIS OF SOME BIOLOGICALLY ACTIVE PYRAZOLE DERIVATIVES

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ABSTRACT
3-(4-Substituted phenyl)-5-phenyl-2,3-dihydro-1*H*-pyrazole (2a-c), 3-(4-substituted phenyl)-2-5-diphenyl-2,3-dihydro-1*H*-pyrazole (3a-c), [5-(4-substituted phenyl)-3-phenyl-2,5-dihydro-1*H*-pyrazole-1-yl](pyridine-4-yl)methanone (4a-c), [5-(4-substituted phenyl)-3-phenyl-2,5-dihydro-1*H*-pyrazole-1-yl](pyridine-3-yl)methanone (5a-c) and 5-(4-substituted phenyl)-3-phenyl-2,5-dihydro-1*H*-pyrazole-1-carbothioamide (6a-c) have been synthesized by microwave assisted synthesis of 3-(4-Substituted phenyl)-1-phenylprop-2-en-1-ones (Chalcones) (1a-c) with hydrazine hydrate, phenyl hydrazine, isoniazide, nicotinic hydrazide or thiosemicarbezide, respectively. The synthesized compounds were characterized by IR and ^{1}H NMR spectral data. Synthesized compounds have been estimated for probable activities using PASS (http://www.195.172.207.233/PASS).
Keywords: Pyrazoles, PASS, Activity prediction, Microwave irradiation.

INTRODUCTION

Pyrazoles and their substituted derivatives are most extensively investigated class of organic compounds and important biological agents. Malladi *et al*.[1] reported the synthesis, characterization and antibacterial activity of some new pyrazole based Schiff bases whereas Hassan *et al*.[2] have synthesized some new pyrazoline and pyrazole derivatives and their antibacterial and antifungal activity. Synthesis and anti tubercular activity of novel 3,5-diaryl-4,5-dihydro-1H-pyrazole derivatives have been studied by Alegaon[3] while Anand *et al*.[4] have carried out the synthesis, antimicrobial and antioxidant activities of some new 3-indolyl pyrazolo[2,3-c]pyran and its derivatives. Novel N-((1,3-substituted diphenyl-1H-pyrazole-4-yl)methylene)-2-methylindoline-1-amine derivatives using MTT method have been synthesized by Thakrar *et al*.[5] and they have also studied their anti-HIV activity. Design, Synthesis and Biological evaluation of Pyrazole analogues of Natural Piperine have been synthesized by Mathew *et. al*.[6] and studied their anti-inflammatory activity. Kalusalingam *et. al*.[7] have Synthesized some pyrazole derivatives and their anti-convulsant activity. Kenichi *et. al*.[8] have reported design, synthesis and pharmacological evaluation of N-bicyclo-5-chloro-1H-indole-2-carboxamide derivatives as potent glycogen phosphorylase inhibitor and anti-diabetic activity while Bhattaglia *et al*.[9] studied indole amide derivatives: synthesis, structure- activity relationships and molecular modellings studies of a new series of histamine H_1-receptor antagonists and anti-histaminic activity. Zalaru *et al*.[10] have synthesized some novel 2-(1*H*-pyrazol-1-yl)-acetamidesas lidocaine analogue and studied their anesthetic activity. Amir and Sahu *et al*.[11,12] reported analgesic activity of pyrazole derivatives whereas Vrma and Kang *et al*.[13,14] studied insecticidal activity of pyrazole derivatives.[15,16]

MATERIALS AND METHODS

All melting points reported are uncorrected and were determined on open capillary tube apparatus. The ^{1}H NMR spectra were recorded in $CDCl_3$/DMSO-d6 solutions on a DRX-300 MHz spectrometer (300 MHz) using TMS as internal standard with chemical shifts are expressed in δ ppm. The IR spectra were measured on a Perkin-Elmer 157 spectrometer using KBr pellets. All the reactions routinely monitored by Thin-layer chromatography (TLC) using silica gel-G. Spots were exposed in an iodine chamber.

General procedure for preparation of chalcone (1a-c)

A convenient route for the synthesis of α, β-unsaturated ketones (Chalcone) was achieved by the reaction of p-substituted benzaldehyde (0.005 mol) with acetophenone (0.005 mol) in the presence of piperidine, under microwave irradiation for 2 minutes. The reaction is being monitored by TLC method. After the completion of reaction, the mixture was quenched with cold water with crushed ice. The extract was washed with distilled water, dried and purified by re crystallization from ethanol.

General procedure for preparation of compound (2a-c), (3a-c), (4a-c), (5a-c) and (6a-c)

A mixture of the chalcone (0.004 mol.), corresponding hydrazines (hydrazine hydrate, phenyl hydrazine, isoniazide, nicotinic hydrazide or thiosemicarbezide) (0.004 mol) in ethanol (10 mL) with glacial acetic acid (2 drop) was irradiated under microwaves for 1 minute. The reaction is being monitored by TLC method (Eluent: $CHCl_3$-MeOH (7:3)). After the completion of reaction, the mixture was quenched with cold water with crushed ice and kept overnight at room temperature. The solid obtained was washed with distilled water and recrystallized from ethanol.

3-(4-Fluorophenyl)-5-phenyl-2, 3-dihydro-1*H*-pyrazole (2a)

Yield 79 %, m.p. 179^{o}C ; IR (KBr) cm^{-1}: 3381, 3411 (N-H pyrazole); 3045 (–Ar-CH); 1350 (C-F str.); ^{1}H NMR (DMSO d_6) δ: 9.98, 9.82 (2H, NH); 4.62 (N-CH); 6.62-7.24 (Ar-H); Anal. Calcd. for $C_{15}H_{13}FN_2$: C, 74.98; H, 5.45; N, 11.66 %. Found: C, 74.59; H, 5.27; N, 11.51 %.

3-(4-Chlorophenyl)-5-phenyl-2, 3-dihydro-1*H*-pyrazole (2b)

Yield 77 %, m.p. 172^{o}C; IR (KBr) cm^{-1}: 3384, 3413 (N-H pyrazole); 3043 (–Ar-CH); 750 (C-Cl str.); ^{1}H NMR (DMSO d_6) δ: 9.96, 9.78 (2H, NH); 4.65 (N-CH); 6.57-7.26 (Ar-H); Anal. Calcd. for $C_{15}H_{13}ClN_2$: C, 70.18; H, 5.10; N, 10.91 %. Found: C, 70.22; H, 5.01; N, 10.82 %.

3-(4-Hydroxyphenyl)-5-phenyl-2, 3-dihydro-1*H*-pyrazole (2c)
Yield 82 %, m.p. 185°C; IR (KBr) cm^{-1}: 3379, 3408 (N-H pyrazole); 3038 (–Ar-CH); 3337 (O-H str.); ^{1}H NMR (DMSO d_6) δ: 9.92, 9.81 (-NH, pyrazole); 4.68 (N-CH); 6.49-7.21 (Ar-H); Anal. Calcd. for $C_{15}H_{14}N_2O$: C, 75.61; H, 5.92; N, 11.76 %. Found: C, 75.45; H, 5.81; N, 11.69 %.

3-(4-Fluorophenyl)-2, 5-diphenyl-2, 3-dihydro-1*H*-pyrazole (3a)
Yield 71 %, m.p. 198°C; IR (KBr) cm^{-1}: 3415 (N-H, pyrazole); 3048 (–Ar-CH); 1342 (C-F str.); ^{1}H NMR (DMSO d_6) δ: 9.76 (NH, pyrazole); 5.42 (N-CH); 6.58-6.99 (Ar-H); Anal. Calcd. For $C_{21}H_{17}FN_2$: C, 79.72; H, 5.42; N, 8.85 %. Found: C, 79.51; H, 5.34; N, 8.81 %.

3-(4-Chlorophenyl)-2, 5-diphenyl-2, 3-dihydro-1*H*-pyrazole (3b)
Yield 76 %, m.p. 193°C; IR (KBr) cm^{-1}: 3421 (N-H, pyrazole); 3052 (–Ar-CH); 762 (C-Cl); ^{1}H NMR (DMSO d_6) δ: 9.72 (NH, pyrazole); 5.58 (N-CH); 6.51-7.05 (Ar-H); Anal. Calcd. for $C_{21}H_{17}ClN_2$: C, 75.78; H, 5.15; N, 8.42 %. Found; C, 75.58; H, 5.12; N, 8.36 %.

3-(4-Hydroxyphenyl)-2, 5-diphenyl-2, 3-dihydro-1*H*-pyrazole (3c)
Yield 73 %, m.p. 187°C; IR (KBr) cm^{-1}: 3426 (N-H pyrazole); 3057 (-Ar-CH); 3342 (O-H str.); ^{1}H NMR (DMSO d_6) δ: 9.73 (NH, pyrazole); 5.52 (N-CH); 6.54-6.98 (Ar-H); Anal. Calcd. for $C_{21}H_{18}N_2O$: C, 80.23; H, 5.77; N, 8.91 %. Found: C, 80.11; H, 5.64; N, 8.88 %.

[5-(4-fluorophenyl)-3-phenyl-2, 5-dihydro-1*H*-pyrazole-1-yl](pyridine-4-yl)methanone (4a)
Yield 82 %, m.p. 169°C; IR (KBr) cm^{-1}: 3421 (N-H pyrazole); 3054 (-Ar-CH); 1595 (C=N); 1654 (C=O); 1352 (C-F str.); ^{1}H NMR (DMSO d_6) δ: 9.72 (NH, pyrazole); 5.54 (N-CH); 6.54-7.28 (Ar-H); Anal. Calcd. for $C_{21}H_{16}FN_3O$: C, 73.03; H, 4.67; N, 12.17 %. Found: C, 73.09; H, 4.54; N, 12.08 %.

[5-(4-Chlorophenyl)-3-phenyl-2, 5-dihydro-1*H*-pyrazole-1-yl](pyridine-4-yl)methanone (4b)
Yield 79 %, m.p. 171°C; IR (KBr) cm^{-1}: 3419 (N-H pyrazole); 3049 (-Ar-CH); 1592 (C=N); 1655 (C=O); 756 (C-Cl str.); ^{1}H NMR (DMSO d_6) δ: 9.74 (NH, pyrazole); 5.52 (N-CH); 6.53-7.29 (Ar-H); Anal. Calcd. for $C_{21}H_{16}ClN_3O$: C, 69.71; H, 4.46; N, 11.61 %. Found: C, 69.47; H, 4.39; N, 11.51 %.

[5-(4-Hydroxyphenyl)-3-phenyl-2, 5-dihydro-1*H*-pyrazole-1-yl](pyridine-4-yl)methanone (4c)
Yield 76 %, m.p. 175°C; IR (KBr) cm^{-1}: 3417 (N-H pyrazole); 3047 (-Ar-CH); 1597 (C=N); 1655 (C=O); 3336 (O-H str.); ^{1}H NMR (DMSO d_6) δ: 9.76 (NH, pyrazole); 5.56 (N-CH); 6.51-7.25 (Ar-H); Anal. Calcd. for $C_{21}H_{17}N_3O_2$: C, 73.45; H, 4.99; N, 12.24 %. Found: C, 73.39; H, 4.85; N, 12.04 %.

[5-(4-Fluorophenyl)-3-phenyl-2, 5-dihydro-1*H*-pyrazole-1-yl](pyridine-3-yl)methanone (5a)
Yield 78 %, m.p. 171°C; IR (KBr) cm^{-1}: 3423 (N-H pyrazole); 3053 (-Ar-CH); 1594 (C=N); 1657 (C=O); 1354 (C-F str.); ^{1}H NMR (DMSO d_6) δ: 9.73 (NH, pyrazole); 5.52 (N-CH); 6.54-7.29 (Ar-H); Anal. Calcd. for $C_{21}H_{16}FN_3O$: C, 73.03; H, 4.67; N, 12.17 %. Found: C, 73.00; H, 4.55; N, 12.04 %.

[5-(4-chlorophenyl)-3-phenyl-2, 5-dihydro-1*H*-pyrazole-1-yl](pyridine-3-yl)methanone (5b)
Yield 76 %, m.p. 172°C; IR (KBr) cm^{-1}: 3421 (N-H pyrazole); 3050 (-Ar-CH); 1591 (C=N); 1656 (C=O); 759 (C-Cl str.); ^{1}H NMR (DMSO d_6) δ: 9.73 (NH, pyrazole); 5.53 (N-CH); 6.51-7.25 (Ar-H); Anal. Calcd. for $C_{21}H_{16}ClN_3O$: C, 69.71; H, 4.46; N, 11.61 %. Found: C, 69.49; H, 4.35; N, 11.54 %.

[5-(4-hydroxyphenyl)-3-phenyl-2, 5-dihydro-1*H*-pyrazole-1-yl](pyridine-3-yl)methanone (5c)
Yield 78 %, m.p. 177°C; IR (KBr) cm^{-1}: 3419 (N-H pyrazole); 3049 (-Ar-CH); 1598 (C=N); 1657 (C=O); 3337 (O-H str.); ^{1}H NMR (DMSO d_6) δ: 9.78 (NH, pyrazole); 5.54 (N-CH); 6.50-7.24 (Ar-H); Anal. Calcd. for $C_{21}H_{17}N_3O_2$: C, 73.45; H, 4.99; N, 12.24 %. Found: C, 73.36; H, 4.81; N, 12.07 %.

5-(4-fluorophenyl)-3-phenyl-2, 5-dihydro-1*H*-pyrazole-1-carbothioamide (6a)
Yield 84 %, m.p. 188°C; IR (KBr) cm^{-1}: 3310 (NH_2), 3375 (N-H pyrazole); 3049 (-Ar-CH); 1243 (C=S); ^{1}H NMR (DMSO d_6) δ: 7.38 (2H, NH_2); 9.79 (NH, pyrazole); 4.67 (N-CH); 6.54-7.31 (Ar-H); Anal. Calcd. for $C_{16}H_{14}FN_3S$: C, 64.19; H, 4.71; N, 14.04 %. Found: C, 64.13; H, 4.65; N, 14.00 %.

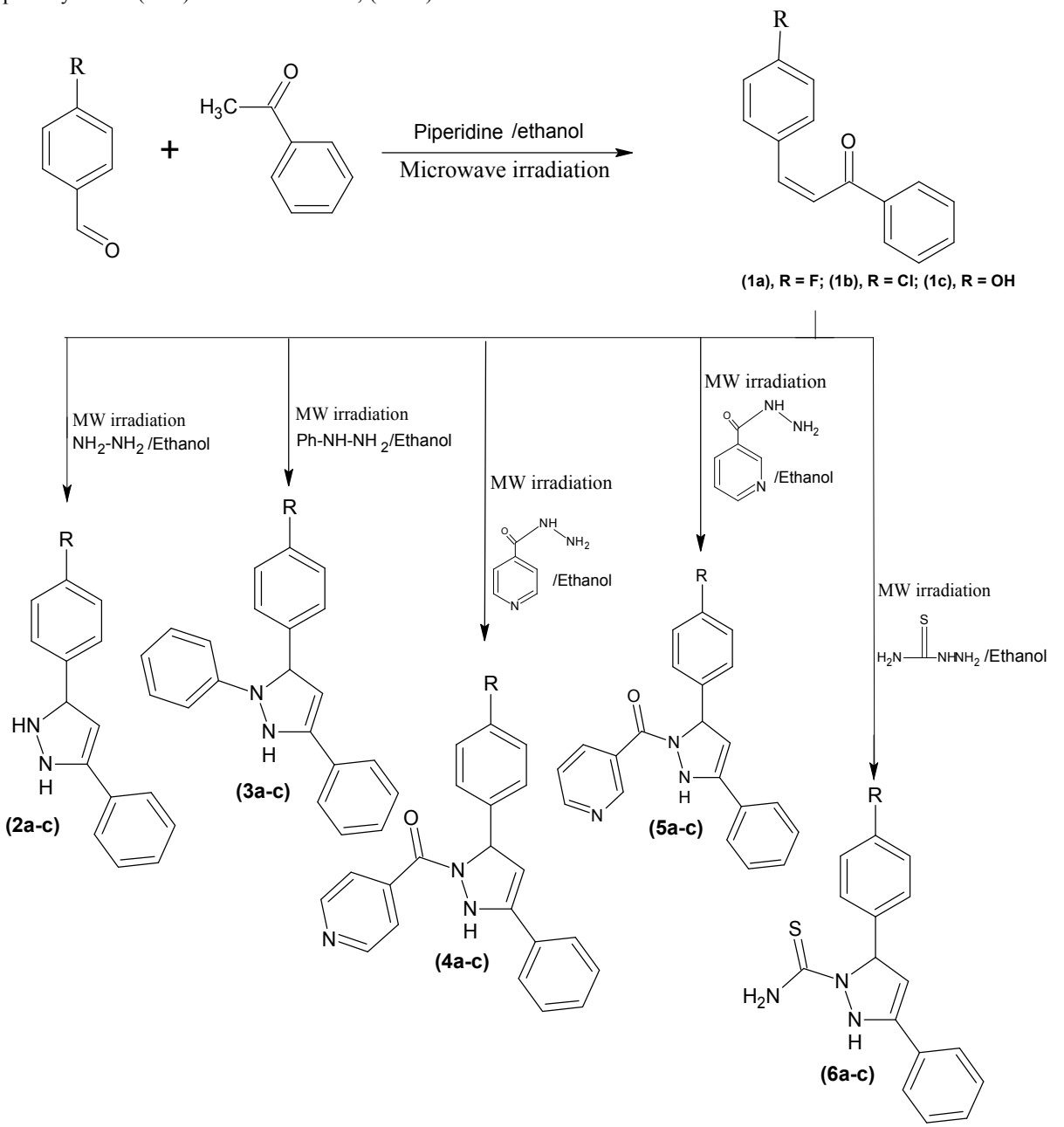
5-(4-chlorophenyl)-3-phenyl-2, 5-dihydro-1*H*-pyrazole-1-carbothioamide (6b)
Yield 79 %, m.p. 176°C; IR (KBr) cm^{-1}: 3308 (NH_2), 3377 (N-H pyrazole); 3052 (-Ar-CH); 1241 (C=S); ^{1}H NMR (DMSO d_6) δ: 7.39 (2H, NH_2); 9.76 (NH, pyrazole); 4.65 (N-CH); 6.56-7.28 (Ar-H); Anal. Calcd. for $C_{16}H_{14}ClN_3S$: C, 60.85; H, 4.47; N, 13.31 %. Found: C, 60.80; H, 4.35; N, 13.24 %.

5-(4-hydroxyphenyl)-3-phenyl-2, 5-dihydro-1*H*-pyrazole-1-carbothioamide (6c)
Yield 74 %, m.p. 168°C; IR (KBr) cm^{-1}: 3313 (NH_2), 3375 (N-H pyrazole); 3046 (-Ar-CH); 1245 (C=S); ^{1}H NMR (DMSO d_6) δ: 7.41 (2H, NH_2); 9.82 (NH, pyrazole); 4.68 (N-CH); 6.53-7.29 (Ar-H); Anal. Calcd. for $C_{16}H_{15}N_3OS$: C, 64.62; H, 5.08; N, 14.13 %. Found: C, 64.53; H, 5.00; N, 14.04 %.

RESULTS AND DISCUSSION

The starting compounds 3-(4-substituted phenyl)-1-phenylprop-2-en-1-ones (Chalcones) (1a-c) react with corresponding hydrazides in ethanol containing a few drops of glacial acetic acid under microwave irradiation to give (2a-c), (3a-c), (4a-c), (5a-c) and (6a-c). The structure was established though IR and ^{1}H NMR spectral data. The IR spectra of (2a-c) exhibited absorption bands for primary amine (-NH) at 3379-3413 cm^{-1}, (–N-N) at 1244- 1248 cm^{-1} and (-C-N) at 1084-1085 cm^{-1}. The ^{1}H NMR spectra of these compounds revealed signals at δ = 9.92 - 9.78 ppm for (-NH) ring proton, a singlet at δ = 4.62-4.68 ppm for (-N-CH) at pyrazole ring, a multiplet at δ = 6.49-7.26 ppm for the aromatic protons. The IR of (3a-c) exhibited absorption bands for primary amine (-NH) at 3415-3426 cm^{-1} and (–N-N) at

1240-1242 cm^{-1} and (-C-N) at 1108-1118 cm^{-1}. The ^1H NMR spectra of these compounds revealed signals at δ = 9.73- 9.76 ppm for (-NH) proton, a singlet at δ = 5.42-5.58 ppm for (-N-CH) at pyrazole ring and a multiplet at δ = 6.51-7.05 ppm for the aromatic proton. The IR of (4a-c) exhibited absorption bands for primary amine (-NH) at 3417-3421 cm^{-1}, (-C=N) at 1592-1597 cm^{-1}, (-C=O) at 1654-1655, (–N-N) at 1241-1245 cm^{-1} and (-C-N) at 1085-1120 cm^{-1}. The ^1H NMR spectra of these compounds revealed signals at δ = 9.72-9.76 ppm for (-NH) ring proton, a singlet at δ = 5.52-5.56 ppm for (-N-CH) at pyrazole ring and a multiplet at δ = 6.51-7.29 ppm for the aromatic proton. The IR of (5a-c) exhibited absorption bands for primary amine (-NH) at 3419-3423 cm^{-1}, (-C=N) at 1591-1598 cm^{-1}, (-C=O) at 1656-1657, (–N-N) at 1242-1244 cm^{-1} and (-C-N) at 1074-1121 cm^{-1}. The ^1H NMR spectra of these compounds revealed signals at δ = 9.73-9.78 ppm for (-NH) proton, a singlet at δ = 5.52-5.54 ppm for (-N-CH) at pyrazole ring and a multiplet at δ = 6.50-7.29 ppm for the aromatic proton. The IR of (6a-c) exhibited absorption bands for primary amine (-NH) at 3375-3377cm^{-1}, 3308-3313 cm^{-1} for ($-NH_2$), 1241-1245 cm^{-1} for (–C=S). The ^1H NMR spectra of these compounds revealed signals at δ = 9.76-9.82 ppm for (-NH) proton, a singlet at δ = 4.65-4.68 ppm for (-N-CH) at pyrazole ring and a multiplet at δ = 6.53-7.31 ppm for the aromatic proton.



Scheme1. Synthesis of compound (2a-c), (3a-c), (4a-c), (5a-c) and (6a-c)

Table 1: Activity prediction data of synthesized compounds

Activity		PKI	SGRKI	TDDI	CYP2A8 substrate	Analgesic	AV	AI	ANP (brain cancer)	Nootropic	ATB
2a	Pa	0.872	0.844	0.624	0.485	0.232	0.358	0.643	0.350	-	-
	Pi	0.002	0.001	0.050	0.037	0.132	0.049	0.053	0.125	-	-
2b	Pa	0.858	0.836	0.822	0.798	0.302	0.492	0.274	0.278	-	0.228
	Pi	0.002	0.001	0.010	0.004	0.037	0.054	0.093	0.167	-	0.134
2c	Pa	0.854	0.836	0.798	-	0.241	0.447	0.409	0.510	-	0.266
	Pi	0.002	0.001	0.013	-	0.115	0.077	0.020	0.068	-	0.094
3a	Pa	0.417	0.417	0.490	0.536	0.797	0.325	0.203	0.344	-	-
	Pi	0.018	0.018	0.092	0.027	0.008	0.190	0.184	0.015	-	-
3b	Pa	0.825	0.374	0.735	0.825	0.778	0.471	0.203	0.368	-	-
	Pi	0.003	0.030	0.024	0.003	0.005	0.064	0.184	0.011	-	-
3c	Pa	0.139	0.409	0.700	0.619	0.632	0.426	0.325	0.363	-	0.220
	Pi	0.017	0.019	0.031	0.016	0.016	0.091	0.054	0.012	-	0.144
4a	Pa	0.111	0.393	0.566	0.515	0.220	0.234	0.557	0.333	0.568	0.212
	Pi	0.030	0.024	0.066	0.031	0.173	0.159	0.090	0.134	0.091	0.154
4b	Pa	0.098	0.357	0.789	0.814	0.243	0.419	0.614	0.175	0.447	0.266
	Pi	0.043	0.337	0.015	0.003	0.111	0.095	0.065	0.097	0.175	0.094
4c	Pa	-	0.388	0.760	0.432	-	0.377	0.663	0.466	0.455	0.313
	Pi	-	0.025	0.020	0.050	-	0.130	0.046	0.088	0.169	0.064
5a	Pa	0.126	0.378	0.423	0.505	0.219	-	0.586	0.330	0.613	-
	Pi	0.021	0.028	0.122	0.033	0.174	-	0.077	0.135	0.071	-
5b	Pa	0.112	-	0.678	0.809	0.229	0.321	0.641	0.284	0.487	-
	Pi	0.030	-	0.036	0.004	0.137	0.195	0.054	0.162	0.141	-
5c	Pa	0.129	0.374	0.639	0.423	-	-	0.225	0.437	0.493	0.245
	Pi	0.021	0.030	0.046	0.052	-	-	0.152	0.091	0.136	0.115
6a	Pa	0.105	0.386	-	-	0.268	0.330	0.290	0.380	-	-
	Pi	0.036	0.026	-	-	0.123	0.067	0.170	0.112	-	-
6b	Pa	-	0.347	0.469	0.601	0.259	0.358	0.259	0.322	-	-
	Pi	-	0.042	0.101	0.018	0.084	0.149	0.202	0.140	-	-
6c	Pa	-	0.381	0.426	-	-	0.349	0.295	0.500	-	0.232
	Pi	-	0.027	0.121	-	-	0.099	0.075	0.072	-	0.131

TDDI: Taurine dehydrogenase inhibitor, SGRKI: Serum-glucocorticoid regulated kinase inhibitor, PKI: Protein kinase inhibitor, AV: Antiviral, AI: Anti-inflammatory, ANP: Anti-neoplastic, ATB: Anti-tuberculosis

Activity prediction

The biological activity of the 3-(4-substituted phenyl)-5-phenyl-2,3-dihydro-1*H*-pyrazole (2a-c), 3-(4-substituted phenyl)-2-5-diphenyl-2,3-dihydro-1*H*-pyrazole (3a-c), [5-(4-substituted phenyl)-3-phenyl-2,5-dihydro-1*H*-pyrazole-1-yl](pyridine-4-yl)methanone (4a-c), [5-(4-substituted phenyl)-3-phenyl-2,5-dihydro-1*H*-pyrazole-1-yl](pyridine-3-yl)methanone (5a-c), 5-(4-substituted phenyl)-3-phenyl-2,5-dihydro-1*H*-pyrazole-1-carbothioamide (6a-c) were obtained by PASS software. The predictions were carried out based on analysis of training set containing about 10000 drugs and biologically active compounds. This set was considered as reference for known chemical compounds as well as different biological activities; percent activity (pa) and inactivity (pi) of compounds (2a-c), (3a-c), (4a-c), (5a-c) and (6a-c) have been represented in Table 1.

CONCLUSION

CADD has considerably extended its range of applications, spanning almost all stages in the drug discovery pipeline, from target identification to lead discovery, from lead optimization to preclinical or clinical trials.

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