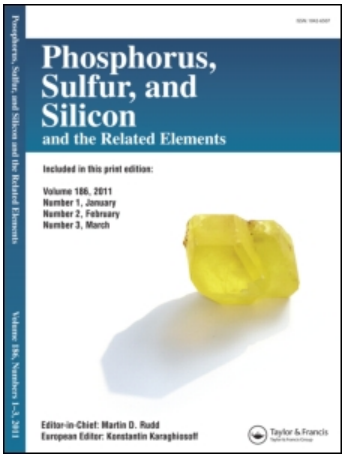

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Synthesis and Spectral Characterization of Novel 1,3,4-Oxadiazole and 1,2,4-Triazole Derivatives: Synthesis for Potential Pharmacological Activities

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SYNTHESIS AND SPECTRAL CHARACTERIZATION OF NOVEL 1,3,4-OXADIAZOLE AND 1,2,4-TRIAZOLE DERIVATIVES: SYNTHESIS FOR POTENTIAL PHARMACOLOGICAL ACTIVITIES

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*Oxadiazole derivatives (**3a**,**b**) and (**4a**,**b**) were obtained in a good yield by the reaction of the benzylidene derivatives (**2a**,**b**) with acetic anhydride and yellow mercuric oxide respectively. Cyclodesulfurization of the thiosemicarbazide derivatives (**5a–c**) with yellow mercuric oxide afforded the oxadiazoles (**7a–c**). On the other hand, reaction of (**5a–c**) with sodium hydroxide gave the triazoles (**6a–c**). The structures of the isolated products were fully determined by spectral methods.*

Keywords: 1,2,4-triazole; 1,3,4-oxadiazole; thiosemicarbazide

INTRODUCTION

It has been reported that disubstituted thiosemicarbazides derivatives undergo oxidative cyclization to oxadiazoles,[1,2] mercaptotriazoles,[3] and thiadiazoles[4] depending on the oxidizing reagent used. Moreover, oxidative cyclization of benzylidene derivatives were also extensively used as a good precursor for the synthesis of oxadiazole derivatives.[5–8] In this study, we have explored the generality of that type of ring closure in the synthesis of new 1,3,4-oxadiazole and 1,2,4-triazole derivatives. 1,3,4-oxadiazoles were reported to show many biological activities for example, bactericide[9] and fungicide[8–10] properties. It is also reported in the literature that some 1,2,4-triazoles have shown biological application such as antiinflammatory,[11] and antihistaminic properties.[12] These

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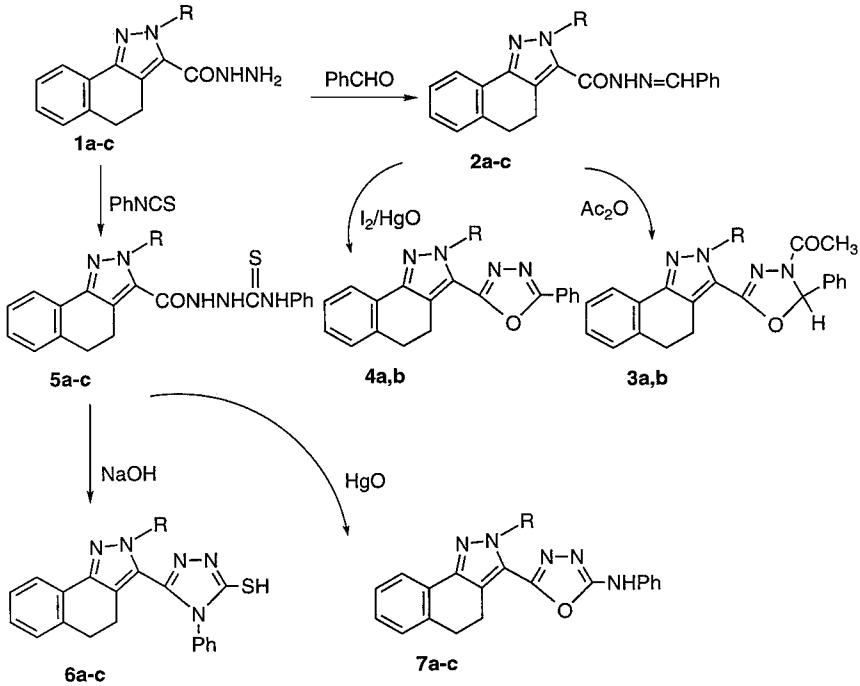
observations, and our interest in the chemistry of oxadiazoles[1,2,13] and triazoles,[13] prompted us to synthesize unreported derivatives of 1,3,4-oxadiazole and 1,2,4-triazole.

DISCUSSION

2-substituted 3-benzylidenehydrazinocarbonyl-4, 5-dihydronaphtho [1,2-*c*]pyrazoles (**2a–c**) for oxadiazoles (**3a**,**b**) and (**4a**,**b**) syntheses were prepared by the condensation of hydrazides derivatives (**1a–c**) with benzaldehyde.[7] On the other hand, thiosemicarbazides (**5a–c**) for triazoles (**6a–c**) and oxadiazoles (**7a–c**) syntheses were prepared by the reaction of hydrazides (**1a–c**) with phenyl isothiocyanate. The results of the reactions are outlined in Scheme 1, Table I and Table II.



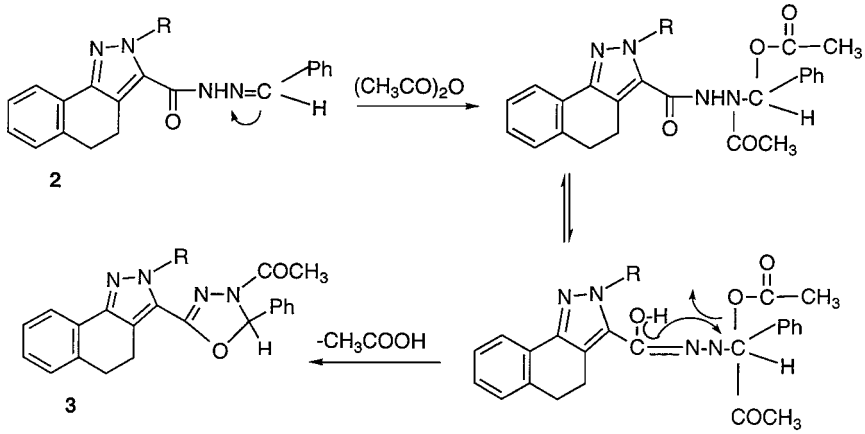
SCHEME 1

The structure of the benzylidene derivatives (**2a–c**) was confirmed from their spectral data. For example, their ^{1}H NMR spectra showed beside the aliphatic, aromatic, and NH protons a singlet of one proton intensity at δ 8.55–8.62 for the =CH. This high deshielded value for the azomethine proton confirm the structure (See Experimental Section).

TABLE I Characterization Data of Benzylidenes (**2a–c**), Oxadiazolines (**3a**,**b**), and Oxadiazoles (**4a**,**b**)

Product	R	Yield (%)	Melting point (°C)	Reaction time (h)
2a	Ph	74	112	3
2b	*p*-ClC_6H_4	57	214	3
2c	*p*-$NH_2SO_2C_6H_4$	69	287	3
3a	Ph	64	86	2
3b	*p*-ClC_6H_4	55	195	2
4a	Ph	60	150	48
4b	*p*-ClC_6H_4	51	172	48

Cyclization of the benzylidenes (**2a**,**b**) with acetic anhydride[5] gave the corresponding 2-aryl-3-(4′-acetyl-5′-phenyl-4′, 5′-dihydro-1′,3′,4′-oxadiazol-2′-yl)-4, 5-dihydronaphtho[1, 2-*c*]pyrazoles (**3a**,**b**). The mechanism of the cyclization reaction[5] is outlined in Scheme 2. The reaction would be initiated by nucleophilic addition of acetic anhydride at the



SCHEME 2

TABLE II Characterization Data of Triazoles (**6a–c**) and Oxadiazolines (**7a–c**)

Product	R	Yield (%)	Melting point (°C)	Reaction time (hours)
6a	Ph	72	138	1
6b	*p*-ClC_6H_4	68	281	1
6c	*p*-$NH_2SO_2C_6H_4$	63	218	1
7a	Ph	61	119	4
7b	*p*-ClC_6H_4	57	167	4
7c	*p*-$NH_2SO_2C_6H_4$	64	260	4

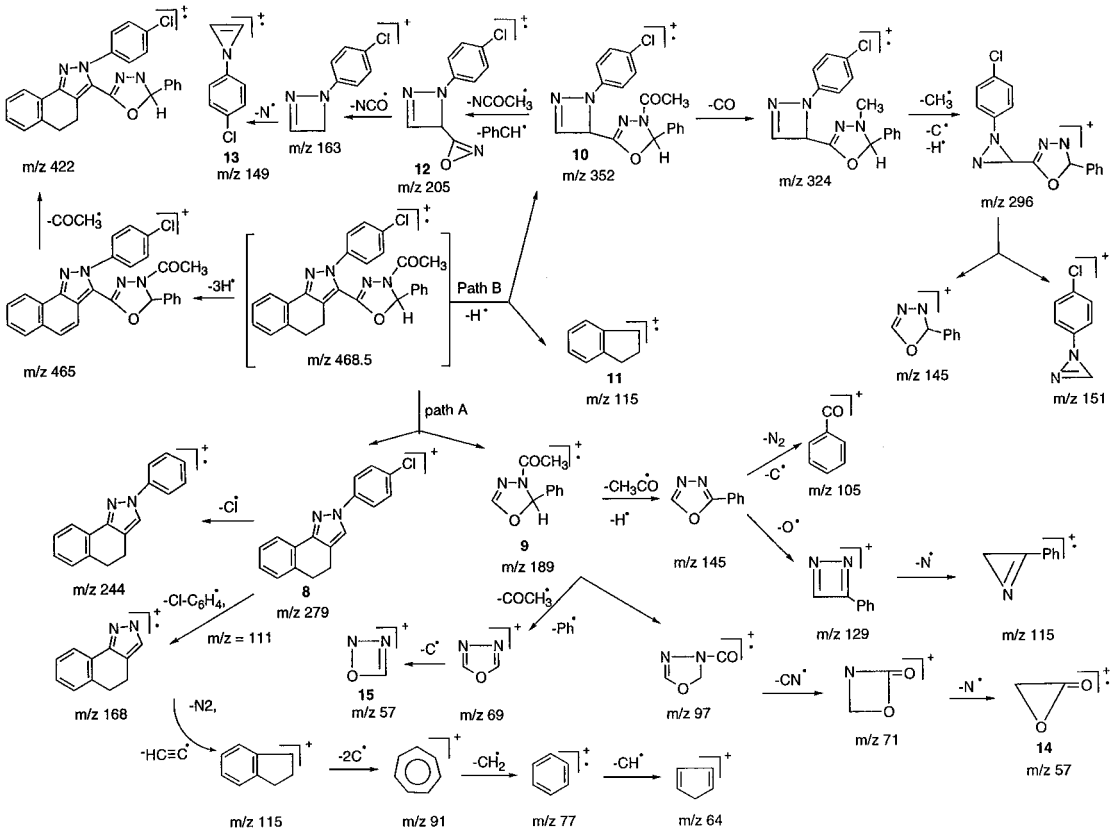
azomethine carbon of the carbazone moiety followed by tautomerization. Subsequent nucleophilic attack by the unshared electron over the oxygen atom of the hydroxyl group at the benzylic carbon and loss of acetic acid afford the desired product (**3a**,**b**).

The infrared spectra of the dihydrooxadiazole derivatives (**3a**,**b**) lacked the NH band but showed a carbonyl absorption at 1667–1668 cm^{-1} for the acetyl group. Their structures were further confirmed from ^1H NMR spectra which lacked the CH= singlet exist in the starting benzylidene and exhibited a singlet of three proton intensity at δ 2.13–2.15 ppm for the $COCH_3$. The ^{13}C NMR spectrum of the N-acetyl oxadiazole derivative (**3b**) exhibited beside the aromatic carbons, three signals at δ 19.99, 29.99, and 92.00 for the C-4, C-5, and CH, respectively as well as a carbonyl carbon at δ 167.83 ppm. These observations are in accordance with mass spectrum of (**3b**). The probable structure of the common prominent peaks as well as their fragmentation pathways are shown in Scheme 3, which does not show molecular ion peak. However, some other fragments are well observed which confirmed its structure. The base peak appeared at m/z 57 and was assigned to the oxiranium or oxadiaztium ions (**14**) or (**15**) respectively. Loss of three hydrogen atoms from the molecular ion gives rise to the weak abundant ion at m/z 465. Fragmentation of the molecular ion according to pathway (A) gives the cation (**8**) and the radical cation (**9**) at m/z 279 and 189, respectively, while loss of hydrogen atom (pathway B) yields the moderately intense radical cations (**10**) and (**11**) at m/z 352 and 115 respectively. Successive elimination of $NCOCH_3$ and PhCH radicals from (**10**) gives rise to the weakly intense peak at m/z 205 formulated as the radical cation (**12**). The strong intense peak at m/z 149 is formulated as the radical cation (**13**) formed by successive loss of NCO and N radicals from (**12**). Other fragmentations are also shown in Scheme 3.

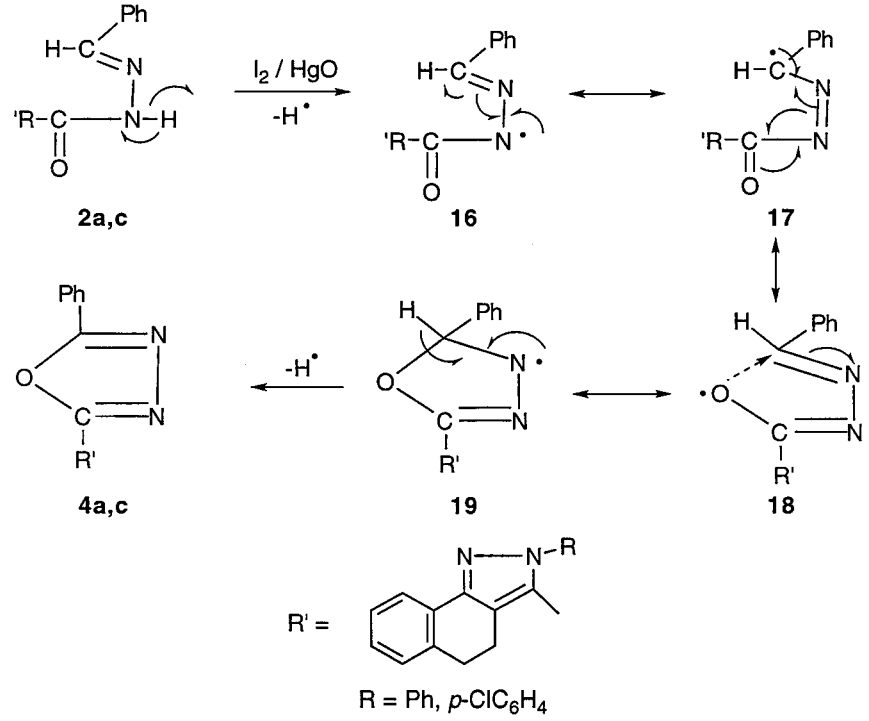
On the other hand, oxidation of benzylidene derivatives (**2a**,**c**) with yellow mercuric oxide and iodine in anhydrous conditions afforded the corresponding oxadiazole derivatives (**4a**,**b**). The plausible mechanism for oxidative cyclization to oxadiazoles (**4**) are outlined in Scheme 4.

The benzylidene derivative (**2**) looses hydrogen atom under the influence of oxidizing I_2/HgO to afford a radical (**16**) which undergoes conjugation to afford radicals (**17**) and (**18**). Cyclization of radical (**18**) with subsequent elimination of another hydrogen atom affords the desired oxadiazole (**4**).

The structure of the oxadiazoles (**4a**,**b**) are supported by the infrared spectra which does not reveal the presence of NH band present in the starting benzylidenes (**2a**,**b**). Moreover, the ^1H NMR spectra does not show the benzylidene as well as the NH protons characteristic for the starting compounds (**2a**,**b**).



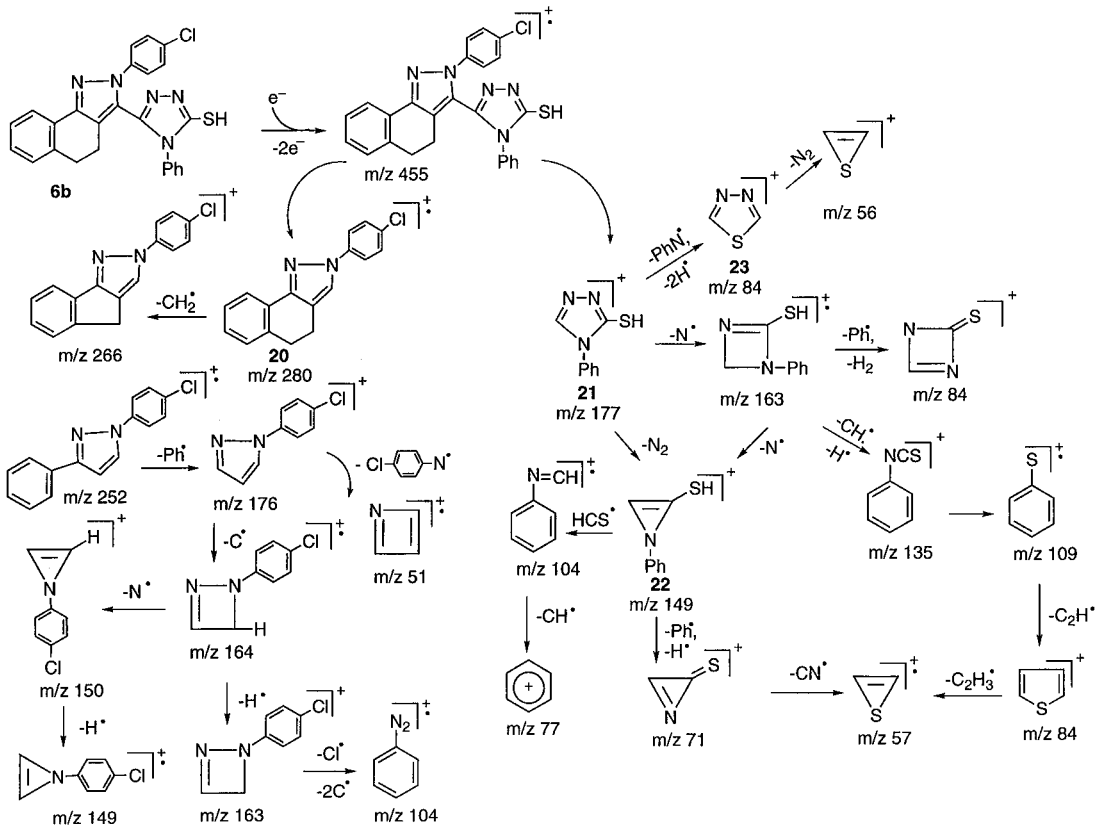
SCHEME 3



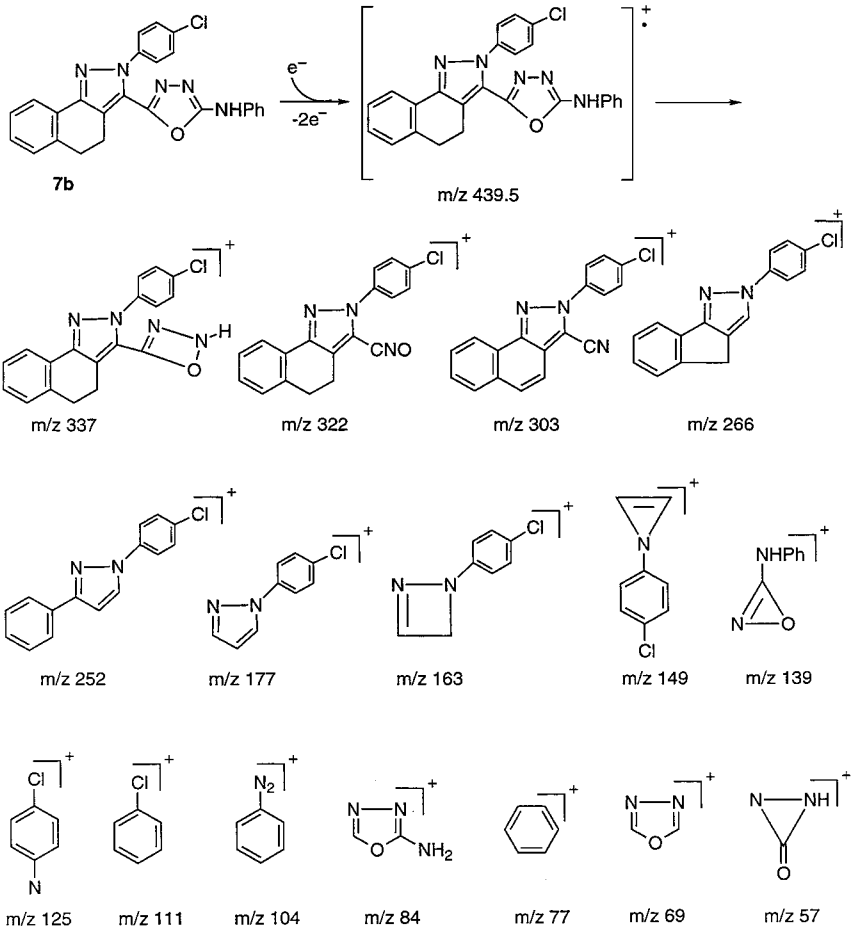
SCHEME 4

Next, treatment of thiosemicarbazide derivatives (**5a–c**)[14] with aqueous sodium hydroxide afforded the desired 1,2,4-triazoles (**6a–c**). The infrared spectra of (**6a–c**) indicating their existance in the thiol form rather than thione one. It showed a SH stretching absorption at 2560–2565 cm^{-1} and lack of any NH stretching. Moreover, their ^{13}C NMR spectra further confirmed the thiol form over the thione one since no C=S signal was appeared in the spectra. The mass spectrum of the triazole derivative (**6b**) showed no molecular ion peak but we could identify some other fragments that confirmed the structure. Fragmentation of the molecular ion gives rise to the species (**20**) and (**21**) at m/z 280 and 177 respectively. Elimination of N_2 molecule from (**21**) leads to the strong intense peak (**22**) at m/z 149, while successive loss of NPh and hydrogen radicals from (**21**) gives rise to the cation (**23**) at m/z 84. The base peak at m/z 163 arises from (**21**) by lossing nitrogen radical. Other fragmentation are outlined in Scheme 5.

On the other hand, treatment of the thiocarbazide derivatives (**5a–c**) with yellow mercuric oxide afforded the corresponding oxadiazoles (**7a–c**) in a good yield. Their structures were assigned on the



SCHEME 5



SCHEME 6

basis of their elemental analyses and spectral data. The infrared spectra revealed a C=N stretching vibrations at 1583–1599 cm^{-1} as well as an NH absorptions at 3255–3349 cm^{-1}. The ^1H NMR showed also an exchangeable NH signal. The structure of the oxadiazole derivatives was further confirmed by mass spectra (Scheme 6).

EXPERIMENTAL

^1H and ^{13}C NMR spectra were recorded on Bruker DPX-400FT NMR spectrometers using TMS as internal standard. Mass spectra were determined on a Kratos MS 30. The infrared spectra were measured on

a Nicolet Magna FT 520 spectrophotometer using potassium bromide pellets. Microanalyses were performed on a 2400 Perkin Elmer Series 2 CHNS analyzer. Melting points were determined on a Gallenkamp melting point apparatus and are uncorrected.

2-phenyl-3-benzylidenehydrazinocarbonyl-4, 5-dihydronaphtho[1,2-*c*]pyrazole (2a)

A solution of benzaldehyde (0.17 g, 1.7 mmol) in ethanol (3 ml) was added to a solution of an equimolar amount of 2-phenyl-4,5-dihydronaphtho[1,2-*c*]pyrazole-3-carbohydrazide (**1a**, 0.5 g, 1.7 mmol) in ethanol (15 ml). The reaction mixture was heated under reflux for 3 h, concentrated, and then left at room temperature overnight. The product which separated was filtered off, washed with ethanol (3 ml), dried, and recrystallized from ethanol to give **2a** as cream needles.; ir (potassium bromide): 1598 (C=N), 1666 (C=O), and 3262 cm^{-1} (NH); ^1H NMR (deuteriochloroform): δ 3.10 (m, 4H, H-4 and H-5), 7.34 (m, ArH and NH), 8.58 (s, 1H, CH=). Anal. Calcd. for C_{25} $H_{20}N_4O$: C, 76.53; H, 5.10; N, 14.29. Found: C, 76.62; H, 5.18; N, 14.38.

2-*p*-chlorophenyl-3-benzylidenehydrazinocarbonyl-4, 5-dihydronaphtho[1,2-*c*]pyrazole (2b)

Compound (**2b**) was obtained as cream crystals from ethanol in a manner similar to the synthesis of (**2a**); ir (potassium bromide): 1544 (C=N), 1666 (C=O), and 3262 cm^{-1} (NH); 1H NMR (DMSO-d_6): δ 3.08 (m, 4H, H-4 and H-5), 7.38 (m, ArH and NH), 8.55 (s, 1H, CH=). Anal. Calcd. for C_{25} $H_{19}ClN_4O$: C, 70.34; H, 4.45; N, 13.13. Found: C, 70.45; H, 4.56; N, 13.34.

2-*p*-sulfamylphenyl-3-benzylidenehydrazinocarbonyl-4, 5-dihydronaphtho[1,2-*c*]pyrazole (2c)

Compound **2c** was obtained as pale yellow crystals from ethanol in a manner similar to the synthesis of (**2a**); ir (potassium bromide): 1345–1166 (SO_2N). 1548 (C=N), 1682 (C=O), and 3331 cm^{-1} (NH); ^1H NMR (DMSO-d_6): δ 3.05 (m, 4H, H-4 and H-5), 7.53 (m, ArH and NH), 8.62 (s, 1H, CH=) Anal. Calcd. for C_{25} $H_{21}N_5O_3S$: C, 63.69; H, 4.46; N, 14.86; S. 6.79. Found: C, 63.81; H, 4.62; N, 14.77; S; 6.89.

2-phenyl-3-(4′-acetyl-5′-phenyl-4′, 5′-dihydro-1′,3′, 4′-oxadiazol-2′-yl)-4,5-dihydronaphtho[1,2-*c*]pyrazole (3a)

A mixture of 2-phenyl-3-benzylidenehydrazinocarbonyl-4,5-dihydronaphtho[1,2-*c*]pyrazole (**2a**) (0.2 g, 0.5 mmol) and acetic anhydride (10 ml) was heated under reflux for 3 h. After the reaction mixture attained room temperature, it was poured into crushed ice and the oily product deposited was decanted from water and extracted with ether. The ether layer was washed with sodium bicarbonate, followed by water, dried over anhydrous sodium sulphate, and evaporated to give (**3a**) as yellow needles.; ir (potassium bromide): 1597 (C=N) and 1668 cm^{-1} (C=O); ^1H NMR (deuteriochloroform): δ 2.15 (s, 3H, CH_3CO), 3.05 (m, 4H, H-4 and H-5), 7.19 (m, ArH and OC*H*Ph). Anal. Calcd. for C_{27} $H_{22}N_4O_2$: C, 74.65; H, 5.07; N, 12.90. Found: C, 74.85; H, 5.22; N, 13.01.

2-*p*-chlorophenyl-3-(4′-acetyl-5′-phenyl-4′, 5′-dihydro-1′,3′, 4′-oxadiazol-2′-yl)-4,5-dihydronaphtho[1,2-*c*] pyrazole (3b)

Compound **2c** was obtained as pale yellow crystals from ethanol in a manner similar to the synthesis of (**3a**); ir (potassium bromide): 1590 (C=N) and 1667 cm^{-1} (C=O); ^1H NMR (deuteriochloroform/DMSO-d_6): δ 2.13 (s, 3H, CH_3CO), 3.08 (m, 4H, H-4, and H-5), 7.14 (m, ArH and OC*H*Ph). Ms: m/z (relative abundance) 465 (1), 422 (2), 352 (21), 324 (1), 296 (2), 279 (25), 244 (4), 205 (4), 189 (2), 168 (4), 163 (3), 151 (10), 149 (85), 145 (3), 129 (11), 115 (10), 105 (21), 97 (23), 91 (5), 77 (6), 71 (37), 69 (51), 64 (8), 57 (100). Anal. Calcd. for C_{27} $H_{21}ClN_4O_2$: C, 69.16; H, 4.48; N, 11.95. Found: C, 69.50; H, 4.82; N, 11.76.

2-phenyl-3-(5′-phenyl-1′,3′,4′-oxadiazol-2′yl)-4, 5-dihydronaphtho[1,2-*c*]-pyrazol (4a)

A mixture of 2-phenyl-3-benzylidenehydrazinocarbonyl-4, 5-dihydronaphtho[1,2-*c*]pyrazole (**2a**, 0.2 g, 0.5 mmol) in dry ether (50 ml) was stirred with yellow mercuric oxide (1.0 g), magnesium oxide (0.4 g) and iodine (1.0 g) at room temperature for 48 h under anhydrous condition. The reaction mixture was filtered off and the ether layer washed with 5% potassium iodide (50 ml), 5% sodium thiosulphate (50 ml), water, dried over anhydrous sodium sulphate, and evaporated to give (**4a**) as yellow needles.; ir (potassium bromide): 1595 cm^{-1} (C=N); ^1H NMR (deuteriochloroform): δ 3.09 (m, 4H, H-4 and H-5), 7.25 (m, ArH). Anal. Calcd. for C_{25} $H_{18}N_4O$: C, 76.92; H, 4.62; N, 14.36. Found: C, 77.13; H, 4.43; N, 14.58.

2-*p*-chlorophenyl-3-(5′-phenyl-1′,3′, 4′-oxadiazol-2′yl)-4, 5-dihydronaphtho-[1,2-*c*]-pyrazol (4b)

Compound (**4b**) was obtained as pale yellow crystals from ethanol in a manner similar to the synthesis of (**4a**); ir (potassium bromide): 1598 cm^{-1} (C=N); ^1H NMR (deuteriochloroform): δ 3.07 (m, 4H, H-4 and H-5), 7.12 (m, ArH). Anal. Calcd. for C_{25} $H_{17}ClN_4O$: C, 70.67; H, 4.00; N, 13.19. Found: C, 70.75; H, 4.11; N, 13.25.

2-phenyl-3-(5′-mercapto-4′-phenyl-1′,2′, 4′-triazol- 3′-yl)-4, 5-dihydronaphtho[1,2-*c*]pyrazol (6a)

A solution of N-phenyl-N′-(2-phenyl-4, 5-dihydronaphtho [1,2-*c*] pyrazol-3-yl-carbonyl)thiosemicarbazide (**5a**) (0.3 g, 0.7 mmol) in 5% sodium hydroxide (5 ml) was heated to reflux for 1 h. The reaction mixture was filtered while hot, cooled, and acidified with dilute HCl to p^H 6. The separated product was filtered off, washed with water till neutral washings, dried, and recrystallized from ethanol-benzene mixture (2:1) to yield (**6a**) as yellow needles.; ir (potassium bromide): 1596 (C=N) and 2565 cm^{-1} (SH); ^1H NMR (deuteriochloroform): δ 3.05 (m, 4H, H-4 and H-5), 7.36 (m, ArH). Anal. Calcd. for C_{25} $H_{19}N_5S$: C, 71.26; H, 4.51; N, 16.60; S, 7.60, Found: C, 71.40; H, 4.46; N, 16.82; S, 7.80.

2-*p*-chlorophenyl-3-(5′-mercapto-4′-phenyl-1′,2′, 4′-triazol-3′-yl)-4,5-dihydronaphtho[1,2-*c*]pyrazol (6b)

Compound (**6b**) was obtained as pale yellow crystals from ethanol-benzene mixture (2:1) in a manner similar to the synthesis of (**6a**); ir (potassium bromide): 1593 (C=N) and 2560 cm^{-1} (SH); ^1H NMR (deuteriochloroform/DMSO-d_6): δ 2.88 (m, 4H, H-4 and H-5), 7.16 (m, ArH). Ms: m/z (relative abundance) 455 (M^+, 1), 280 (1), 266 (69, 252 (2), 177 (1), 164 (11), 163 (11), 150 (1), 149 (21), 135 (4), 109 (2), 104 (7), 84 (6), 77 (14), 71 (9), 57 (19), 56 (10). Anal. Calcd. for C_{25} $H_{18}N_5S$: C, 65.86; H, 3.95; N, 15.37; S, 7.03. Found: C, 65.96; H, 4.00; N, 15.28; S, 7.20.

2-*p*-chlorophenyl-3-(5′-mercapto-4′-phenyl-1′,2′, 4′-triazol-3′-yl)-4,5-dihydronaphtho[1,2-*c*]pyrazol (6c)

Compound (**6c**) was obtained as cream needles from ethanol-benzene mixture (2:1) in a manner similar to the synthesis of (**6a**); ir (potassium bromide): 1332, 1160 (SO_2N), 1594 (C=N) and 2575 cm^{-1} (SH); ^1H NMR (deuteriochloroform/DMSO-d_6): δ 3.06 (m, 4H, H-4 and H-5), 7.45 (m, ArH). Anal. Calcd. for C_{25} $H_{20}N_6O_2S_2$: C, 60.00; H, 4.00; N, 16.80; S, 12.80. Found: C, 60.12; H, 3.95; N, 16.61; S, 12.45.

2-phenyl-3-(5′-phenylamino-1′,3′, 4′-oxadiazol-2′-yl)-4, 5-dihydronaphtho[1,2-*c*]pyrazole (7a)

Finally powdered yellow mercuric oxide (0.18 g, 0.8 mmol) was added portionwise over a period of 30 min to a boiling solution of N-phenyl-N′-(2-phenyl-4,5-dihydronaphtho[1,2-*c*]pyrazol-3-yl-carbonyl)thiosemicarbazide (**5a**) (0.3 g, 0.7 mmol) in ethanol (20 ml). The suspension was stirred and heated to reflux for 4 h and then filtered. The black precipitate (HgS) formed was washed with boiling ethanol (10 ml). The combined filtrate and washings were concentrated and set aside overnight at room temperature. The separated product were recrystallized from ethanol to yield **7a** as cream needles.; ir (potassium bromide): 1598 (C=N) and 3279 cm^{-1} (NH). Anal. Calcd. for C_{25} $H_{19}N_5O$: C, 74.07; H, 4.69; N, 17.28. Found: C, 73.96; H, 4.40; N, 17.50.

2-*p*-chlorophenyl-3-(5′phenylamino-1′,3′,4′-oxadiazol-2′-yl)-4,5-dihydronaphtho[1,2-*c*]pyrazole (7b)

Compound (**7b**) was obtained as cream needles from ethanol in a manner similar to the synthesis of (**7a**); ir (potassium bromide): 1599 (C=N) and 3255 cm^{-1} (NH); ^{1}H NMR (deuteriochloroform): δ 3.08 (m, 4H, H-4 and H-5), 7.27 (m, ArH), 9.52 (broad s, 1H, NH). Ms: m/z (relative abundance) 337 (1), 322 (1), 303 (1), 266 (6), 252 (2), 177 (2), 163 (100), 149 (13), 139 (1), 125 (1), 111 (1), 104 (7), 84 (7), 77 (14), 69 (9), 57 (21). Anal. Calcd. for C_{25} $H_{18}N_5O$: C, 68.26; H, 4.10; N, 15.93. Found: C, 68.67; H, 3.98; N, 16.02.

2-*p*-sulfamylphenyl-3-(5′-phenylamino-1′,3′,4′-oxadiazol-2′-yl)-4,5-dihydronaphtho [1,2-*c*]pyrazole (7c)

Compound (**7c**) was obtained as cream needles from ethanol in a manner similar to the synthesis of (**7a**); ir (potassium bromide): 1330, 1164 (SO_2N), 1583 (C=N), and 3265, 3344 cm^{-1} (NH and NH_2); ^{1}H NMR (DMSO-d_6): δ 3.10 (m, 4H, H-4 and H-5), 7.37 (m, ArH and NH_2), 9.62 (broad s, 1H, NH). Anal. Calcd. for C_{25} $H_{20}N_6O_3$: C, 61.98; H, 4.13; N, 17.63; S, 6.61. Found: C, 61.32; H, 3.93; N, 16.95; S, 6.43.

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