Microwave Assisted Synthesis of Novel 1,2,3,4-Tetrahydrocarbazolyl thiazolidin-4-ones and Azetidin-2-ones and its Biological Behavior

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Received 11 January 2009; Accepted 6 March 2009

Abstract: A new family of 1,2,3,4 – tetrahydrocarbazolyl thiazolidin-4-ones (**4a-e**) and 1,2,3,4-tetrahydrocarbazolyl azetidin-2-ones (**5a-e**) were individually derived using *N*-[(α -substituted bezylidenehydrazino) acetyl] -1,2,3,4-tetrahydrocarbazoles **3a-e** by cyclization with thioglycolic acid and chloroacetylchloride respectively. The compounds (**3a-e**) were prepared by condensation of *N*-(hydrazinoacetyl) 1,2,3,4-tetrahydrocarbazole (**2**) with a series of aldehydes. The compound **2** was obtained by the hydrazinolysis of *N*-(chloroacetyl)-1,2,3,4-tetrahydrocarbazole (**1**). These products were characterized by IR, NMR, MASS spectra and by elemental analysis. All the titled compounds (**4a-e**) and **5(a-e)** were screened for antibacterial and antifungal activities.

Keywords: Microwave assisted synthesis, Thiazolidin-4-ones, Azetidin-2-ones, Antibacterial and antifungal activities.

Introduction

Carbazole derivatives are important class of heterocyclic compounds possessing variety of biological activity¹⁻². Carbazole skeleton bearing natural products fused with heterocyclic ring have drawn significant attention due to excellent pharmacological activities of their

analogues³⁻⁴. There are numerous evidences illustrating that the fused ring of heterocycles at Nth position have gained unique importance on pharmacological studies⁵⁻⁶. Azetidinone derivatives have found to possess a wide spectrum of biological activity⁷. Thiazolidinone derivatives are also reported for antibacterial⁸ and antitubercular⁹⁻¹⁰ activities. Keeping this in mind, it was worthwhile to develop the synthesis of title compounds *i.e.* 1,2,3,4-tetrahydrocarbazolyl azetidinones and tetrahydrocarbazolyl thiazolidinones. Moreover, the microwave assisted reactions¹¹ using dry media¹² have attracted much interest because of the simplicity in operation, greater selectivity and rapid synthesis of a variety of heterocyclic compounds¹³.

The synthetic route for the conventional methodology is depicted in Scheme 1. *N*-(chloroacetyl)-1,2,3,4-tetrahydrocarbazole (**1**) was prepared by the acylation of 1,2,3,4-tetrahydrocarbazole¹⁴ with chloroacetyl chloride in dry DMF in presence of anhydrous potassium carbonate at ambient temperature. The hydrazinolysis of compound (**1**) with hydrazine hydrate gave *N*-(hydrazinoacetyl)-1,2,3,4-tetrahydrocarbazole (**2**). Condensation of compound (**2**) with various aromatic aldehydes in DMF gave corresponding hydrazones (**3a-e**). Cyclization of (**3a-e**) with thioglycolic acid in DMF containing anhydrous zinc chloride and chloroacetylchloride in dioxane containing triethylamine afforded tetrahydrocarbazolyl thiazolidinones (**4a-e**) and tetrahydro carbazolyl azetidinones (**5a-e**) respectively. The homogeneity of the compounds was checked by TLC (ethylacetate: hexane 50:50). All the synthesized compounds were characterized by elemental analysis, IR, NMR and mass spectrometric techniques (Table 1). Compounds *N*-(chloroacetyl)-1,2,3,4-tetrahydrocarbazole (**1**) and *N*-(hydrazinoacetyl)-1,2,3,4-tetrahydro carbazole (**2**) were prepared as per the procedure found in literature¹⁵. The antibacterial activities of the title compounds (**4a-e**) and (**5a-e**) were evaluated against the organisms.

Scheme 1. Synthesis of 1,2,3,4-tetrahydrocarbazolyl thiazolidin-4-ones and azetidin-2-ones.

Table 1. Analytical data of (4a-e) and (5a-e).

Compd. No.	Yield, %	Melting point °C	Molecular formula	Elemental analysis, %		
				Carbon Calc/Found	Hydrogen Calc/Found	Nitrogen Calc/Found
1	83	95	C ₁₄ H ₁₄ ClNO	67.80/67.17	5.70/5.62	5.65/5.28
2	80	126	C ₁₄ H ₁₇ N ₃ O	69.11/69.35	7.04/7.73	17.27/17.20
3a	88	182	C ₂₁ H ₂₁ N ₃ O	76.11/76.21	6.39/6.50	12.68/12.33
3b	90	214	C ₂₁ H ₂₁ N ₃ O ₂	72.60/72.34	6.09/6.70	12.10/12.14
3c	84	168	C ₂₁ H ₂₀ N ₄ O ₃	67.01/67.20	5.36/5.62	14.88/14.28
3d	89	172	C ₂₂ H ₂₃ N ₃ O	76.44/76.32	6.71/6.78	12.16/12.34
3e	91	152	C ₂₂ H ₂₃ N ₃ O ₂	73.11/72.33	6.41/6.52	11.63/11.54
4a	77	185	C ₂₄ H ₂₃ N ₃ O ₂ S	68.12/68.24	5.72/5.46	10.36/10.36
4b	84	181	C ₂₃ H ₂₃ N ₃ O ₃ S	65.54/65.72	5.50/5.52	9.97/9.82
4c	80	230	C ₂₃ H ₂₂ N ₄ O ₄ S	61.32/61.44	4.92/4.62	12.44/12.48
4d	89	217	C ₂₄ H ₂₅ N ₃ O ₂ S	68.71/68.75	6.01/6.11	10.02/10.23
4e	84	230	C ₂₄ H ₂₅ N ₃ O ₃ S	66.18/66.40	5.79/5.68	9.65/9.60
5a	81	210	C ₂₃ H ₂₂ ClN ₃ O ₂	67.73/67.25	5.44/5.80	10.30/10.25
5b	86	220	C ₂₃ H ₂₂ ClN ₃ O ₃	65.17/65.20	5.23/5.35	9.91/9.84
5c	84	212	C ₂₃ H ₂₁ ClN ₄ O ₄	61.00/61.05	4.67/4.72	12.37/12.13
5d	82	231	C ₂₄ H ₂₄ ClN ₃ O ₂	68.32/68.40	5.73/5.35	9.96/9.27
5e	89	181	C ₂₄ H ₂₄ ClN ₃ O ₃	65.82/65.52	5.52/5.42	9.60/9.46

Antimicrobial studies

Newly synthesized compounds (4a-e) and (5a-e) were screened for their *in vitro* antibacterial activity against *Bacillus cereus*, *Bacillus subtilis*, *Streptococci mutans*, and *Micrococcus luteus*, at concentration of 25 µg using ciprofloxacin as standard and antifungal activity against *Aspergillus niger*, *Candida albicans*, *Alternaria macrospora* and *Fusarium oxysporum* at concentration of 25 µg using ketoconazole as standard. DMF was used as solvent control, nutrient agar was used as culture medium and method employed was cup plate method¹⁶. The zones of inhibition formed were measured in mm and are shown in Tables 2 & 3 respectively to antibacterial and antifungal activities.

Table 2. Antibacterial activity of compounds (4a-e) and (5a-e) (zone inhibition in mm)

Compound	<i>B.cereus</i>	<i>B.subtilis</i>	<i>S.aureus</i>	<i>M.luteus</i>
4a	18	18	17	19
4b	27	29	28	25
4c	23	22	24	25
4d	23	26	21	24
4e	25	27	24	26
5a	17	18	16	17
5b	25	24	22	25
5c	21	27	21	25
5d	20	19	17	21
5e	22	21	19	24
Ref. Std	30	32	28	28

DMF-negative control; Reference Standard ciprofloxacin.

Table 3. Antifungal activity of compounds (**4a-e**) and (**5a-e**) (zone inhibition in mm).

Compound	<i>A.niger</i>	<i>C.albicans</i>	<i>F.oxysporum</i>	<i>A.macrospora</i>
4a	17	19	18	15
4b	27	26	28	26
4c	19	20	18	17
4d	24	23	21	23
4e	26	24	28	26
5a	16	18	17	15
5b	25	24	22	24
5c	18	18	20	21
5d	23	22	21	22
5e	24	23	21	21
Ref. Std	28	30	30	28

DMF-negative control; Reference Standard Ketoconazole

Experimental

All melting points were taken by open capillary method and are uncorrected. TLC analysis were done on glass plates coated with Silicagel-G and spotting was done using iodine. IR (KBr) spectra were recorded on Jasco FT-IR 5300 spectrometer. ^1H and ^{13}C NMR spectra were in CDCl_3 using Jeol GSX 400 (400 MHz) and Jeol ECA500 (500 MHz) NMR spectrometer. Mass spectra were recorded using Joel GC mate and MAL-DI-TOF LD spectrometer. Column chromatography was performed using silica gel (100-200 mesh).

Synthesis of N-(α -substituted bezylidenehydrazino acetyl) -1,2,3,4-tetrahydrocarbazole (3a-e)

Compound (**2**) (0.01 mole), a pinch of *p*-toluene sulphonic acid and appropriate aromatic aldehydes (**a-e**) (0.01 mole) were mixed. Acidic alumina was added to the above mixture at room temperature. The reaction mixture was adsorbed, dried and kept inside the alumina bath and irradiated for 40-80 s. The mixture was cooled and the products was extracted with dry methanol and poured into crushed ice. The solid thus separated was filtered, washed thoroughly with water and recrystallized from ethanol to furnish (**3a-e**).

Synthesis of 1,2,3,4 – tetrahydrocarbazolyl thiazolidin-4-ones (4a-e)

Schiff bases (**3a-g**) (0.01 mole) was added to mercaptoacetic acid (1.40 mL, 0.02 mole) anhydrous aluminium chloride (0.05 g). Acidic alumina was added to the above solution at room temperature. The reaction mixture was mixed for 40-80 s. The reaction mixture was then cooled and triturated with an excess of 10% sodium bicarbonate solution. The product obtained was filtered, washed in several times with water and crystallized with isopropanol. Other thiazolidin-4-one (**4a-e**) were synthesized using the similar procedure.

3-(2-(1,2,3,4-Tetrahydrocarbazolyl)-2-oxoethylamino)-2-phenylthiazolidin-4-one (4a)

IR (KBr) :1480,1680,2850 cm^{-1} ; ^1H NMR δ : 1.68-1.72 (m,4H) 2.06-2.12 (m,4H), 2.73 (b,1H, N-H), 3.94 (d,2H), 4.45 (s,2H,-NH-CH₂-) 4.78 (s,1H), 6.44-6.74 (m,4H,ArH), 6.82-7.26 (m,5H, ArH) ^{13}C NMR δ : 21.8, 25.2, 26.2, 33.5, 37.9, 49.8, 111.1, 119.0, 120.2, 122.2, 126.8, 127.0, 127.4,128.6,136.2. MS (EI):*m/z* 405.17[M⁺].

3-(2-(1,2,3,4-Tetrahydrocarbazolyl)-2-oxoethylamino)-2-(4-hydroxyphenyl)thiazolidin-4-one(4b)

IR (KBr) :1490,1685,2852 cm^{-1} ; ^1H NMR δ : 1.67-1.71 (m,4H) 2.00-2.11 (m,4H), 2.72 (b,1H, N-H), 3.97 (d,2H), 4.43(s,2H,-NH-CH₂-) 4.76 (s,1H), 5.20 (s,1H), 6.42-6.73 (m,4H,ArH), 6.81-7.24 (m,4H,ArH) ^{13}C NMR δ : 22.3, 24.0, 27.1, 31.4, 48.4, 110.2, 117.1, 123.1, 124.2, 126.1, 126.2, 129.3,139.1. MS(EI): m/z 421.19[M⁺].

3-(2-(1,2,3,4-Tetrahydrocarbazolyl)-2-oxoethylamino)-2-(2-nitrophenyl)thiazolidin-4-one(4c)

IR (KBr) :1460,1670,2830 cm^{-1} ; ^1H NMR δ : 1.68-1.72 (m,4H) 2.02-2.12 (m,4H),2.74 (b,1H, N-H), 3.72 (bs,3H) 3.96 (d,2H), 4.48 (s,2H,-NH-CH₂-) 4.78 (s,1H),7.42-7.73 (m,4H, ArH), 7.81-7.98 (m, 4H, ArH) ^{13}C NMR δ : 24.6, 25.0, 29.2, 31.5, 49.2, 114.3, 118.4, 123.4, 126.2, 127.5, 130.5, 140.1. MS(EI): m/z 450.19[M⁺].

3-(2-(1,2,3,4-Tetrahydrocarbazolyl)-2-oxoethylamino)-2-(tolyl)thiazolidin-4-one(4d)

IR (KBr) :1458,1660,2840 cm^{-1} ; ^1H NMR δ : 1.63-1.76 (m,4H) 2.04-2.10 (m,4H),2.71(b,1H, N-H), 3.92 (d,2H), 4.40 (s,2H,-NH-CH₂-) 4.72 (s,1H), 6.82-7.24 (m,4H,ArH),7.51-7.76 (m, 4 H , ArH) ^{13}C NMR δ : 20.5, 25.6, 27.1, 35.5, 46.5, 104.3, 119.4, 120.4, 126.3, 130.5, 131.6, 148.1. MS(EI): m/z 448.79[M⁺].

3-(2-(1,2,3,4-Tetrahydrocarbazolyl)-2-oxoethylamino)-2-(4-methoxyphenyl)thiazolidin-4-one(4e)

IR (KBr) :1430,1685,2855 cm^{-1} ; ^1H NMR δ : 1.62-1.75 (m,4H) 2.10-2.12 (m, 4H), 2.68 (bs,3H),2.72 (b,1H, N-H), 3.37(d,2H),3.90(d,2H),4.41(s,2H,-NH-CH₂-)4.73(s,1H),6.78-7.30 (m, 4H, ArH), 7.30-7.40(m,4H,ArH) ^{13}C NMR δ : 21.5, 23.0, 28.1, 36.5, 42.2, 100.1, 117.4, 122.4, 124.4, 132.5, 136.6, 149.2. MS(EI): m/z 436.78[M⁺].

Synthesis of 1,2,3,4 – tetrahydrocarbazolyl azetidin-2-ones(5a-e)

Schiff bases (**3a-g**) (0.01 mole) was mixed with triethylmaine (2.80 mL, 0.02 mole) and chloroacetyl chloride (1.60 mL, 0.02 mole) was added dropwise over a period of 30 min. Acidic alumina was added to the above solution at room temperature. The reaction mixture was added to the above solution at room temperature. The reaction mixture was adsorbed, dried and kept inside the alumina bath and irradiated for 40-80 s. The mixture was cooled and the product was extracted with absolute ethanol and poured into crushed ice. The solid thus separated was filtered, washed thoroughly with water and recrystallized from aqueous ethanol. Other azetidine 2 -ones (**5a-e**) were derived by using the similar procedure.

1-(2-(1,2,3,4-Tetrahydrocarbazolyl)-2-oxoethylamino)-3-chloro-4-phenylazetidin-2-one(5a)

IR (KBr) :1680, 1710 cm^{-1} ; ^1H NMR δ : 1.56-2.43 (m,8H) 2.59(s,1H),3.76 (d, 2H), 5.0 (bs,1H) 5.43 (s,1H),6.92-7.08 (m,5H,-ArH) 7.12-7.28 (m,4H,ArH). ^{13}C NMR δ : 23.5, 23.9, 25.2, 62.2, 50.5, 64.4, 111.1, 120.1, 122.2, 127.4, 136.6, 163.5, 200.0, MS(EI): m/z 436.78[M⁺].

1-(2-(1,2,3,4-Tetrahydrocarbazolyl)-2-oxoethylamino)-3-chloro-4-(4-hydroxyphenyl)azetidin-2-one(5b)

IR(KBr):1675,1708 cm^{-1} ; ^1H NMR δ :1.58-2.46 (m, 8H)2.62 (s,1H),3.68(d, 2H),4.98(bs,1H),5.2 (s,1H),5.45(d,1H), 6.68-6.95 (m,4H,-ArH) 7.18-7.26(m,4H,ArH). ^{13}C NMR δ : 23.6, 23.8, 25.3, 64.1, 51.3, 64.4, 113.1, 115.7, 120.1, 122.2, 127.4, 136.6, 156.5, 162.5, 202.0. MS(EI): m/z 423.78[M⁺].

1-(2-(1,2,3,4-Tetrahydrocarbazolyl)-2-oxoethylamino)-3-chloro-4-(2-nitrophenyl)azetidin-2-one (5c)

IR (KBr): 1640, 1700 cm^{-1} ; ^1H NMR δ : 1.55-2.32 (m, 8H), 2.60 (s, 2H), 3.72 (d, 1H), 5.0 (bs, 1H), 5.44 (s, 1H), 6.68-7.20 (m, 4H, ArH), 7.38-8.14 (m, 4H, ArH) ^{13}C NMR δ : 23.1, 23.6, 25.2, 63.4, 53.6, 64.4, 110.1, 114.2, 121.9, 123.2, 127.2, 136.0, 147.2, 201.4. MS(EI): m/z 452.80 [M^+]

1-(2-(1,2,3,4-Tetrahydrocarbazolyl)-2-oxoethylamino)-3-chloro-4-tolylazetidin-2-one (5d)

IR (KBr) : 1660, 1720 cm^{-1} ; ^1H NMR δ : 1.56-2.43 (m, 8H), 2.62 (s, 2H), 2.86 (bs, 3H), 3.74 (d, 1H), 5.31 (bs, 1H), 5.32 (s, 1H), 6.54-6.94 (m, 4H, ArH), 6.86-7.23 (m, 4H, ArH) ^{13}C NMR δ : 19.0, 22.0, 24.3, 62.2, 51.4, 66.2, 112.0, 115.3, 122.3, 124.0, 127.3, 135.2, 146.2, 204.2. MS(EI): m/z 421.72 [M^+].

1-(2-(1,2,3,4-Tetrahydrocarbazolyl)-2-oxoethylamino)-3-chloro-4-(4-methoxyphenyl)azetidin-2-one (5e)

IR (KBr) : 1650, 1700 cm^{-1} ; ^1H NMR δ : 1.51-2.40 (m, 8H), 2.60 (s, 2H), 3.71 (d, 1H), 3.86 (bs, 3H), 5.30 (bs, 1H), 5.34 (s, 1H), 6.28-6.68 (m, 4H, ArH), 6.82-7.24 (m, 4H, ArH) ^{13}C NMR δ : 20.2, 23.2, 61.0, 52.2, 51.4, 62.4, 112.2, 113.2, 121.1, 122.1, 123.1, 132.7, 158.9, 204.2. MS(EI): m/z 437.82 [M^+].

Results and Discussion

Acylation of 1, 2, 3, 4-tetrahydrocarbazole at N^{th} position was carried out by treating chloroacetyl chloride with 1,2,3,4-tetrahydrocarbazole. The N-H displacement of carbazole with acyl chloride of chloroacetyl chloride was confirmed by the ^1H NMR spectrum of compound (**1**) showed a singlet for CH_2 at δ 4.49 ppm and the disappearance of N-H proton at δ 8.23 ppm in 1,2,3,4-tetrahydrocarbazole. The disappearance of IR bands at 3410 cm^{-1} for N-H stretching vibration in 1,2,3,4-tetrahydrocarbazole and appearance of new band at 1700 cm^{-1} confirmed that the acetylation has been occurred at N^{th} place of 1,2,3,4-tetrahydrocarbazole. The hydrazinolysis of compound (**1**) with hydrazine hydrate afforded *N*-(hydrazinoacetyl)-1, 2, 3, 4-tetrahydrocarbazole (**2**). The ^1H NMR spectrum of hydrazide (**2**), showed a singlet for N-H at δ 3.1 ppm and singlet for NH_2 δ 2.90 ppm. Its IR spectrum showed a band between 3094 and 3316 cm^{-1} . Treatment of compound (**2**) with benzaldehyde in DMF formed *N*-(hydrazinoacetyl)-1,2,3,4-tetrahydrocarbazole (**3a**). The ^1H NMR spectrum displayed a singlet at δ 8.11 for $\text{CH}=\text{N}$ and multiplet between δ 6.82 and δ 7.26 ppm confirmed that the formation of *N*-(hydrazinoacetyl)-1,2,3,4-tetrahydrocarbazole (**3a**).

When *N*-(α -benzylidenehydrazino acetyl)-1,2,3,4-tetrahydrocarbazole (**3a**) underwent cyclization with thioglycolic acid afforded 3-(2-(1,2,3,4-tetrahydrocarbazolyl)-2-oxoethylamino)-2-phenylthiazolidin-4-one (**4a**). In its ^1H NMR spectrum, the appearance of thiazolidinone methylene protons at δ 3.94 ppm and methene proton at δ 4.78 ppm confirmed the structure (**4a**). Its IR spectrum showed a band at 1680 and 2850 cm^{-1} respectively CH_2 and carbonyl groups in thiazolidinone moiety of compound (**4a**). The compound (**3a**) also underwent cyclization with chloroacetyl chloride resulted **5a**. Its IR spectrum showed a band at 1710 cm^{-1} for the formation of β -lactum. The ^1H NMR spectrum displayed a broad singlet at δ 5.0 and δ 5.4 ppm respectively *N*-CH-Ar and highly deshielded $-\text{Cl}-\text{CH}-\text{C}=\text{O}$ protons.

Antibacterial activities of all the newly prepared compounds against four bacteria are presented in Table 2. The antibacterial activity of compounds **(4b)**, **(5b)**, **(4e)** and **(5e)** are respectively having hydroxyl and methoxy groups in heterocyclic moieties found to be excellent. The compounds **(4d)** and **(5d)** are having methyl group exhibited better activity than compounds **(4a)** and **(5a)** and **(4c)** and **(5c)** are respectively having unsubstituted phenyl and nitrophenyl groups in heterocyclic moieties. (Table 2).

Antifungal activities of all the newly prepared compounds against fungi are presented in Table 2. The antifungal activity of compounds **(4b)** and **(4e)** are excellent activity towards *Fusarium oxysporum* and *Alternaria* macrospore among the four organisms. The compounds **(4c)** and **(5c)** and **(4d)** and **(5d)** are having moderate activity where as **(4a)** and **(5a)** are having considerable activities for the tested organisms (Table 3)

Acknowledgement

The authors are grateful to Dr. Ceeal Analytical Lab, Thorappakkam, Chennai for providing antimicrobial studies. The authors also grateful to SAIF, IIT, Chennai for providing spectral data.

References

1. Itoigawa M, Kashiwada Y, Ito C, Furukawa H, Tachibana Y, Bastow K F and Lee K H, *J Nat Prod*, 2000, **63**, 893.
2. Tachibana Y, Kikuzaki H, Lajis N H and Nakatani N, *J Agric Food Chem*, 2001, **49**, 5589.
3. Haider N, *J Heterocyclic Chem.*, 2002, **39**, 511.
4. Arputharaj Ebenezer Martin and Karnam Jayarampillai Rajendra Prasad, *Acta Pharma.*, 2006, **56**, 79.
5. Mogilahiah K and Sakram B, *Indian J Chem.*, 2004, **43B**, 2724.
6. Trivedi S, Kubavat H, and Parekh H, *Indian J Chem.*, 1994, **33B**, 295.
7. Riaz F Abdullah and Kenneth H Fuhr, *J Med Chem.*, 1975, **18(6)**, 625.
8. Hogle M B, Uthale A C and Nikam B P, *Indian J Chem.*, 1991, **30B**, 717.
9. Desai N C, Astik R R and Thaker K A, *J Indian Chem Soc.*, 1982, **59**, 771.
10. Ajay Kumar, Sanjay Malhotra, Archana Vats, Himanshu, Sanjay K Singh, Sunil K Sharma, Ashok K Prasad, William Errington, Carl E Olsen, Subhash C Jain and Virinder S Parmar, *Indian J Chem.*, 2002, **41B**, 360.
11. Villemin D, Martin B and Garrus B, *Synth Commun.*, 1993, **23**, 2251.
12. Kidwai M, Bhushan K R and Kumar P, *Monatsh Chemie.*, 1999, **130**, 585.
13. Kidwai M and Negi N, *Acat Pharm.*, 1995, **45**, 511.
14. Murugavel S, Kannan P S, SubbiahPandi A, Surendiran T and Balasubramanian S *Acta Cryst.*, 2008, **E64**, o2433.
15. Pandey V K, Gupta, Mrinalini Upadhyay, Mridula Updhyay, Singh V K and Meenal Thandon, *Indian J Chem.*, 2005, **44B**, 158-162.
16. Seeley H W and Van Denmark P J, *Microbes in Action: A Laboratory Manual of Microbiology*, Taraporewala, D B Sons and Copt Ltd, 1975, 55.