Arabian Journal of Chemistry (2011) **4**, 179–184

King Saud University

Arabian Journal of Chemistry

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ORIGINAL ARTICLE

Facile synthesis of new 1,2,3-benzotriazolo-2-oxo-azetidine analogues by microwave irradiation

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Received 16 June 2010; accepted 17 June 2010
Available online 11 July 2010

KEYWORDS

Microwave synthesis of 1,2,3-benzotriazole; Azetidinone

Abstract A series of new N^1-[3-{(4-subtitutedaryl-3-chloro-2-oxo-azetidine)-carbamyl}-propyl]-1,2,3-benzotriazoles **4(a–s)** have been synthesized from 1,2,3-benzotriazole as a starting material by microwave method. The structure of all the synthesized compounds were confirmed by chemical and spectral analyses such as IR, ^{1}H NMR, ^{13}C NMR and FAB-Mass.

1. Introduction

Heterocycles are very important class of chemistry and interesting field for research. This field has great opportunities for the synthesis of novel drugs. Here we are reporting the synthesis of a series of 2-oxo-azetidine derivatives of 1,2,3-benzotriazole. 2-oxo-azetidine ring shows various biological activities such as antifungal (Shukla and Srivastava, 2008; Rawat and Srivastava, 1998; Chavan and Pai, 2007; Singh et al., 2007), antibacterial (Nema and Srivastava, 2007; Mulwad et al., 2008; Van der Steen and Koten, 1991; Singh, 2004), antitubercular (Parikh et al., 2000, 2005; Patel et al., 2006), anticonvulsant (Srivastava et al., 2000), analgesic, anti-inflammatory (Srivastava et al., 1999), synthetic precursor for amino acids (Alonsodel et al., 2002), antiviral (Skiles and McNeil, 1990) etc.

Benzotriazole derivatives have broad range of biological activities such as antibacterial (Asati et al., 2006), antifungal (Nema and Srivastava, 2007; Lebouvier et al., 2006), antihistaminic, antiadrenergic and analgesic (Caliendo et al., 1995), DNA cleavage (Annapukowska et al., 2006), antitubercular (Sanna et al., 2000). Now a days microwave synthesis is suitable and favorite technique of researcher for synthesis because it is eco-friendly, control pollution, converts hours into minutes, enhance yield and stop wastage of solvents so it has economically importance and also help to stop global warming. The structures of all the newly synthesized compounds were confirmed by elemental analysis IR, ^{1}H NMR, ^{13}C NMR and FAB-Mass.

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1878-5352

Peer review under responsibility of King Saud University.
doi:10.1016/j.arabjc.2010.06.035

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Table 1 Spectral data of compounds **1**, **2**, **3(a–s)** and **4(a–s)**.

Comp.	IR (cm^{-1})	^{1}H NMR	^{13}C NMR	FAB mass
1	745 (C–Cl), 1232 (N–CH_2), 1560 (C═C), 3018, 2838 (CH)	1.81 (p, 2H, J = 7.45 Hz, $CH_2CH_2CH_2$), 3.44 (t, 2H, J = 7.45 Hz, $CH_2CH_2CH_2$–Cl), 3.72 (t, 2H, J = 7.45 Hz, N–$CH_2CH_2CH_2$), 7.26–7.95 (m, 4H, Ar)	36.4 ($CH_2CH_2CH_2$), 43.8 ($CH_2CH_2CH_2$–Cl), 49.1 (N–$CH_2CH_2CH_2$), 118.7, 120, 128.1, 128.7, 145, 147.7 (6C, Ar)	196M$^+$
2	1230 (C–N), 1652 (C═O), 3348 (NH), 3430 (NH_2)	1.71 (p, 2H, J = 7.42 Hz, $CH_2CH_2CH_2$), 3.28 (t, 2H, J = 7.42 Hz, $CH_2CH_2CH_2$–NH), 3.81 (t, 2H, J = 7.42 Hz, N–$CH_2CH_2CH_2$), 5.60 (s, 1H, NH), 5.88 (s, 2NH_2), 6.86–7.72 (m, 4H, Ar)	39.7 ($CH_2CH_2CH_2$), 43.1 ($CH_2CH_2CH_2$NH), 48.7 (N–$CH_2CH_2CH_2$), 163.4 (CO), 117.9, 121.1, 127.7, 128.2, 144.7, 46.8 (6C, Ar)	219M$^+$
3a	1551 (N═CH), 1660 (C═O), 3362 (NH)	1.64 (p, 2H, J = 7.35Hz, $CH_2CH_2CH_2$), 3.32 (t, 2H, J = 7.35Hz, $CH_2CH_2CH_2$–NH), 3.72 (t, 2H, J = 7.35Hz, N–$CH_2CH_2CH_2$), 5.76 (s, 1H, NH), 7.84 (s, 1H, N═CH), 7.40–7.71 (m, 9H, Ar)	38.2 ($CH_2CH_2CH_2$), 45.2 ($CH_2CH_2CH_2$–N), 51.1 (N–$CH_2CH_2CH_2$), 145 (N═CH), 162.7 (CO), 120, 120.9, 125.7, 126, 127.1, 128.3, 129, 129.2, 131.2, 138, 146, 149 (12C, Ar)	307M$^+$
3b	738 (C–Cl), 1562 (N═CH), 1670 (C═O), 3370 (NH)	1.76 (p, 2H, J = 7.35 Hz, $CH_2CH_2CH_2$), 3.38 (t, 2H, J = 7.35 Hz, $CH_2CH_2CH_2$–NH), 3.81 (t, 2H, J = 7.35 Hz, N–$CH_2CH_2CH_2$), 5.75 (s, 1H, NH), 7.91 (s, 1H, N═CH), 7.28–7.81 (m, 8H, Ar)	38.9 ($CH_2CH_2CH_2$), 44.4 ($CH_2CH_2CH_2$–NH), 47.1 (N–$CH_2CH_2CH_2$), 152 (N═CH), 166 (CO), 120.4, 127.1, 127.7, 128.8, 129, 130, 131, 131.2, 133.3, 137.8, 143.1, 144.2 (12C, Ar)	341M$^+$
3c	736 (C–Cl), 1559 (N═CH), 1673 (C═O), 3365 (NH)	1.73 (p, 2H, J = 7.38 Hz, $CH_2CH_2CH_2$), 3.36 (t, 2H, J = 7.38 Hz, $CH_2CH_2CH_2$–NH), 3.79 (t, 2H, J = 7.38 Hz, N–$CH_2CH_2CH_2$), 5.77 (s, 1H, NH), 7.97 (s, 1H, N═CH), 7.1–7.9 (m, 8H, Ar)	38.5 ($CH_2CH_2CH_2$), 43.5 ($CH_2CH_2CH_2$–N), 52 (N–$CH_2CH_2CH_2$), 158.2 (N═CH), 167.6 (CO), 117, 120, 125.3, 126.4, 128.3, 129.4, 131, 132.5, 135.6, 140, 146.1, 144.9 (12C, Ar)	341M$^+$
3d	730 (C–Cl), 1566 (N═CH), 1673 (C═O), 3371 (NH)	1.69 (p, 2H, J = 7.40 Hz, $CH_2CH_2CH_2$), 3.30 (t, 2H, J = 7.40 Hz, $CH_2CH_2CH_2$–NH), 3.84 (t, 2H, J = 7.40 Hz, N–$CH_2CH_2CH_2$), 5.73 (s, 1H, NH), 8.07 (s, 1H N═CH), 7.2–7.9 (m, 8H, Ar)	38.7 ($CH_2CH_2CH_2$), 44.2 ($CH_2CH_2CH_2$–N), 50 (N–$CH_2CH_2CH_2$), 155.6 (N═CH), 166.9 (CO), 116.4, 122.8, 125, 126.7, 128, 129.2, 130.5, 131, 134, 137.8, 141.2, 147.2 (12C, Ar)	341M$^+$
3e	636 (C-Br), 1562 (N═CH), 1667 (C═O), 3374 (NH)	1.67 (p, 2H, J = 7.50 Hz, $CH_2CH_2CH_2$), 3.31 (t, 2H, J = 7.50 Hz, $CH_2CH_2CH_2$–NH), 3.79 (t, 2H, J = 7.50 Hz, N–$CH_2CH_2CH_2$), 5.78 (s, 1H, NH), 7.97 (s, 1H, N═CH) 7.39–7.68 (m, 8H, Ar)	36.1 ($CH_2CH_2CH_2$), 44.2 ($CH_2CH_2CH_2$–NH), 50.2 (N–$CH_2CH_2CH_2$), 154 (N═CH), 163.1 (CO), 119, 121, 124, 127, 128.4, 129, 130, 134, 139, 141.3, 148, 150 (12C, Ar)	386M$^+$
3f	641 (C–Br), 1562 (N═CH), 1661 (C═O), 3366 (NH)	1.64 (p, 2H, J = 7.52 Hz, $CH_2CH_2CH_2$), 3.27 (t, 2H, J = 7.52 Hz, $CH_2CH_2CH_2$–NH), 3.83 (t, 2H, J = 7.52 Hz, N–$CH_2CH_2CH_2$), 5.72 (s, 1H, NH), 7.98 (s, 1H, N═CH) 7.23–7.9 (m, 8H, Ar)	36.3 ($CH_2CH_2CH_2$), 44.4 ($CH_2CH_2CH_2$–N), 48 (N$CH_2CH_2CH_2$), 151.6 (N═CH), 163.9 (CO), 116, 119.6, 125.2, 126.8, 128, 129.3, 131.1, 136, 138.6, 143.1, 147.3, 152 (12C, Ar)	386M$^+$
3g	628 (C–Br), 1558 (N═CH), 1672 (C═O), 3367 (NH)	1.71 (p, 2H, J = 7.55 Hz, $CH_2CH_2CH_2$), 3.31 (t, 2H, J = 7.55 Hz, $CH_2CH_2CH_2$–NH), 3.77 (t, 2H, J = 7.55 Hz, N–$CH_2CH_2CH_2$), 5.69 (s, 1H, NH), 8.02 (s, 1H, N═CH), 7.31–7.63 (m, 8H, Ar)	38.5 ($CH_2CH_2CH_2$), 44.1 ($CH_2CH_2CH_2$–NH), 51.4 (N–$CH_2CH_2CH_2$), 152 (N═CH), 164.1 (CO), 117.4, 121.5, 125.7, 127, 128, 129.4, 132, 137, 1140.2, 143.3, 147, 151 (12C, Ar)	386M$^+$
3h	847 (C–N), 1528 (N═O), 1568 (N═CH), 1638 (C═O), 3358 (NH)	1.77 (p, 2H, J = 7.62 Hz, $CH_2CH_2CH_2$), 3.29 (t, 2H, J = 7.62 Hz, $CH_2CH_2CH_2$–NH), 3.84 (t,2H, J = 7.62 Hz, N–$CH_2CH_2CH_2$), 5.81 (s, 1H, NH), 8.10 (s, 1H, N═CH), 7.32–7.91 (m, 8H, Ar)	40.7 ($CH_2CH_2CH_2$), 45.3 ($CH_2CH_2CH_2$–N), 50.3 (N$CH_2CH_2CH_2$), 155.9 (N═CH), 162.4 (CO), 109, 110, 120.4, 121, 123, 128, 133, 135.6, 138.1, 141, 144, 149 (12C, Ar)	352M$^+$
3i	848 (C–N), 1524 (N═O), 1572 (N═CH), 1635 (C═O), 3351 (NH)	1.68 (p, 2H, J = 7.60 Hz, $CH_2CH_2CH_2$), 3.16 (t, 2H, J = 7.60 Hz, $CH_2CH_2CH_2$–NH), 3.72 (t, 2H, J = 7.60 Hz, N–$CH_2CH_2CH_2$), 5.82 (s, 1H, NH), 8.08 (s, 1H, N═CH), 7.21–7.86 (m, 8H, Ar)	40.7 ($CH_2CH_2CH_2$), 44.2 ($CH_2CH_2CH_2$–NH), 49.1 (N–$CH_2CH_2CH_2$), 154.7 (N═CH), 163.2 (CO), 111, 114, 119.4, 121.4, 124, 129, 133.3, 135, 138.5, 140, 145, 151 (12C, Ar)	352M$^+$
3j	842 (C–N), 1531 (N═O), 1575 (N═CH), 1644 (C═O), 3351 (NH)	1.74 (p, 2H, J = 7.60 Hz, $CH_2CH_2CH_2$), 3.25 (t, 2H, J = 7.60 Hz, $CH_2CH_2CH_2$–NH), 3.72 (t, 2H, J = 7.60 Hz, N–$CH_2CH_2CH_2$), 5.83 (s, 1H, NH), 8.17 (s, 1H, N═CH), 7.26–7.99 (m, 8H, Ar)	40.1 ($CH_2CH_2CH_2$), 45.1 ($CH_2CH_2CH_2$–N), 48.7 (N$CH_2CH_2CH_2$), 155.1 (N═CH), 162 (CO), 110.2, 115, 120, 121.6, 125, 126, 132, 135.5, 138.9, 142, 144.2, 151 (12C, Ar)	352M$^+$
3k	1561 (N═CH), 2945 (OCH_3), 3351 (NH)	1.71 (p, 2H, J = 7.55 Hz, $CH_2CH_2CH_2$), 3.28 (t, 2H, J = 7.55 Hz, $CH_2CH_2CH_2$–NH), 3.50 (s, 3H, OCH_3), 3.63 (t, 2H, J = 7.55 Hz, N$CH_2CH_2CH_2$), 5.71 (s, 1H, NH), 7.85 (s, 1H, N═CH), 7.34–7.52 (m, 8H, Ar)	37.2 ($CH_2CH_2CH_2$), 42.6 ($CH_2CH_2CH_2$–NH), 47 (N–$CH_2CH_2CH_2$), 51.7 (OCH_3), 154.2 (N═CH), 160.5 (CO), 111.3, 114.5, 115, 118, 119.6, 122, 128, 131, 135.1, 146, 151, 158 (12C, Ar)	337M$^+$
3l	1557 (N═CH), 2943 (OCH_3), 3358 (NH)	1.68 (p, 2H, J = 7.60 Hz, $CH_2CH_2CH_2$), 3.31 (t, 2H, J = 7.60 Hz, $CH_2CH_2CH_2$–NH), 3.61 (s, 3H, OCH_3), 3.68 (t, 2H, J = 7.60 Hz, N–$CH_2CH_2CH_2$), 5.69 (s, 1H NH), 7.96 (s, 1H, N═CH), 7.41–7.82 (m, 8H, Ar)	37.7 ($CH_2CH_2CH_2$), 42.9 ($CH_2CH_2CH_2$–NH), 47.7 (N–$CH_2CH_2CH_2$), 54.7 (OCH_3), 153.7 (N═CH), 161.9 (CO), 110.2, 114.1, 115.4, 117, 119, 120.3, 129.7, 132, 136.4, 142, 148, 158.7 (12C, Ar)	337M$^+$
3m	1559 (N═CH), 2947 (OCH_3), 3361 (NH)	1.72 (p, 2H, J = 7.60 Hz, $CH_2CH_2CH_2$), 3.30 (t, 2H, J = 7.60 Hz, $CH_2CH_2CH_2$–NH), 3.67 (s, 3H, OCH_3), 3.75 (t, 2H, J = 7.60 Hz, N–$CH_2CH_2CH_2$), 5.74 (s, 1H, NH), 7.86 (s, 1H, N═CH), 7.22–7.72 (m, 8H, Ar)	38.1 ($CH_2CH_2CH_2$), 43.1 ($CH_2CH_2CH_2$–NH), 53.7 (OCH_3), 48.1 (N–$CH_2CH_2CH_2$), 151 (N═CH), 158.1 (CO), 113.2, 114.8, 117.4, 118, 119.5, 122, 127.7, 132.6, 135, 145, 147.4, 157.3 (12C, Ar)	337M$^+$

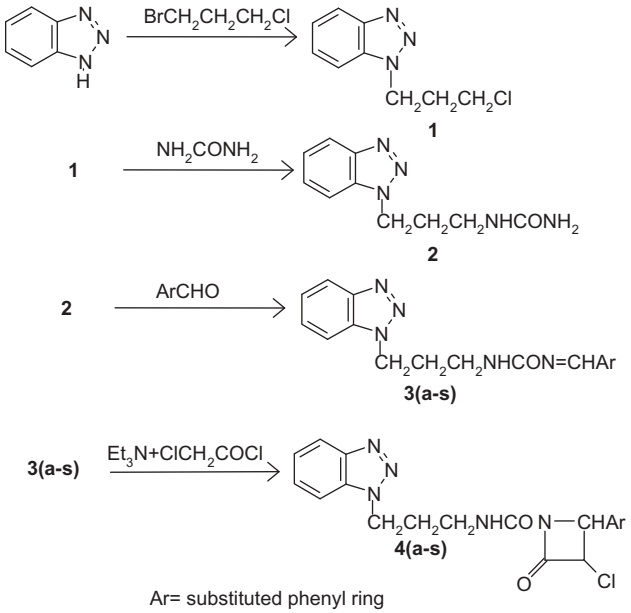
Table 1 *(continued)*

Comp.	IR (cm^{-1})	^1H NMR	^{13}C NMR	FAB mass
3n	1549 (N═CH), 2917 (CH_3), 3340 (NH)	1.61 (p, 2H, J = 7.40 Hz, $CH_2CH_2CH_2$), 2.64 (s, 3H, CH_3), 3.22 (t, 2H, J = 7.40 Hz, $CH_2CH_2CH_2$–NH), 3.72 (t, 2H, J = 7.40 Hz, N–$CH_2CH_2CH_2$), 5.62 (s, 1H, NH), 7.89 (s, 1H, N═CH), 7.39–7.79 (m, 8H, Ar)	24.9 (CH_3), 36.6 ($CH_2CH_2CH_2$), 42.4 ($CH_2CH_2CH_2$–NH), 46.7 (N–$CH_2CH_2CH_2$), 151.2 (N═CH), 160.8 (CO), 120, 121.5, 123, 127.3, 128.9, 130.7, 131.7, 136, 138, 142.1, 145, 146.3 (12C, Ar)	321M^+
3o	1544 (N═CH), 2921 (CH_3), 3345 (NH)	1.63 (p, 2H, J = 7.42 Hz, $CH_2CH_2CH_2$), 2.58 (s, 3H, CH_3), 3.13 (t, 2H, J = 7.42 Hz, $CH_2CH_2CH_2$–NH), 3.75 (t, 2H, J = 7.42 Hz, N–$CH_2CH_2CH_2$), 5.69 (s, 1H, NH), 7.91 (s, 1H, N═CH), 7.31–7.83 (m, 8H, Ar)	22.9 (CH_3), 36.7 ($CH_2CH_2CH_2$), 42.1 ($CH_2CH_2CH_2$–NH), 45.7 (N–$CH_2CH_2CH_2$), 152 (N═CH), 159.8 (CO), 118, 121, 125, 126, 128.1, 130.2, 132, 136.6, 140, 143, 144.5, 147 (12C, Ar)	321M^+
3p	1551 (N═CH), 2908 (CH_3), 3341 (NH)	1.65 (p, 2H, J = 7.40 Hz, $CH_2CH_2CH_2$), 2.60 (s, 3H, CH_3), 3.24 (t, 2H, J = 7.40 Hz, $CH_2CH_2CH_2$–NH), 3.64 (t, 2H, J = 7.40 Hz, N–$CH_2CH_2CH_2$), 5.62 (s, 1H, NH), 7.88 (s, 1H, N═CH), 7.34–7.76 (m, 8H, Ar)	21.9 (CH_3), 38.2 ($CH_2CH_2CH_2$), 43.3 ($CH_2CH_2CH_2$–NH), 45.7 (N–$CH_2CH_2CH_2$), 154 (N═CH), 161.2 (CO), 119, 120.5, 124.5, 126.1, 126.1, 131.9, 131.7, 135, 139, 143, 144, 145 (12C, Ar)	321M^+
3q	1557 (N═CH), 3385 (NH), 3472 (OH)	1.80 (p, 2H, J = 7.65 Hz, $CH_2CH_2CH_2$), 3.37 (t, 2H, J = 7.65 Hz, $CH_2CH_2CH_2$–NH), 3.71 (t, 2H, J = 7.65 Hz, N–$CH_2CH_2CH_2$), 4.15 (s, 1H, OH), 5.82 (s, 1H, NH), 8.07 (s, 1H, N═CH), 7.32–7.79 (m, 8H, Ar)	39.9 ($CH_2CH_2CH_2$), 45.3 ($CH_2CH_2CH_2$–NH), 50.1 (N–$CH_2CH_2CH_2$), 153.3 (N═CH), 166.7 (CO), 117, 118.9, 120.7, 122.7, 125.9, 128.4, 129.8, 132.2, 142.3, 146.9, 147.1, 154.6 (12C, Ar)	323M^+
3r	1561 (N═CH), 3379 (NH), 3464 (OH)	1.78 (p, 2H, J = 7.68 Hz, $CH_2CH_2CH_2$), 3.39 (t, 2H, J = 7.68 Hz, $CH_2CH_2CH_2$–NH), 3.81 (t, 2H, J = 7.68 Hz, N–$CH_2CH_2CH_2$), 4.26 (s, 1H, OH), 5.86 (s, 1H, NH), 8.01 (s, 1H, N═CH), 7.36–7.74 (m, 8H, Ar)	40 ($CH_2CH_2CH_2$), 44.7 ($CH_2CH_2CH_2$–N), 49.7 (N–$CH_2CH_2CH_2$), 151 (N═CH), 165.5 (CO), 114, 116, 121, 123, 125, 128.4, 132.8, 138.4, 143, 146, 147.1, 151.6 (12C, Ar)	323M^+
3s	1567 (N═CH), 3381 (NH), 3468 (OH)	1.74 (p, 2H, J = 7.70 Hz, $CH_2CH_2CH_2$), 3.34 (t, 2H, J = 7.70 Hz, $CH_2CH_2CH_2$–NH), 3.76 (t, 2H, J = 7.70 Hz, N–$CH_2CH_2CH_2$), 4.36 (s, 1H, OH), 5.83 (s, 1H, NH), 7.97 (s, 1H, N═CH), 7.25–7.69 (m, 8H, Ar)	38.4 ($CH_2CH_2CH_2$), 43.3 ($CH_2CH_2CH_2$–NH), 49.1 (N–$CH_2CH_2CH_2$), 151.3 (N═CH), 164.1 (CO), 114, 116.5, 123.7, 125.7, 126.9, 130.4, 130.8, 133.5, 140.3, 145.9, 147.8, 154 (12C, Ar)	323M^+
4a	1327 (C–N), 1730 (CO cyclic), 2908 (CH–Cl)	4.46 (d, J = 5.0 Hz, 1H, CH–Cl), 5.19 (d, J = 5.0 Hz, 1H, N–CH), 5.62 (s, 1H, NH), 6.85-7.72 (m, 9H, Ar)	54.2 (CH–Cl), 62.4 (N–CH), 159.8 (NHCO), 163.7 (CO cyclic), 117, 118.9, 121.7, 122.4, 128.4, 128.8, 129.1, 130.2, 131, 142.3, 145.9, 147.1 (12C, Ar)	384M^+
4b	762 (C–Cl), 1338 (C–N), 1750 (C═O cyclic), 2910 (CH–Cl)	4.62 (d, 1H, J = 5.12 Hz, CH–Cl), 5.36 (d, 1H, J = 5.12 Hz, N–CH), 5.87 (s, 1H, NH), 6.86–7.72 (m, 8H, Ar)	53.8 (CH–Cl), 63.4 (N–CH), 166.2 (CO, cyclic), 116, 118.9, 120.7, 122.7, 125.6, 128.4, 129.8, 132.2, 135.4, 142.3, 146.9, 147.1 (12C, Ar)	418M^+
4c	774 (C–Cl), 1333 (C–N), 1755 (CO cyclic), 2918 (CH–Cl)	4.68 (d, 1H, J = 5.10 Hz, CH–Cl), 5.36 (d, 1H, J = 5.10 Hz, N–CH), 5.84 (s, 1H, NH), 6.79–7.64 (m, 8H, Ar)	55.8 (CH–Cl), 65.1 (N–CH), 165.5 (CO cyclic), 115, 118, 120.1, 123.7, 126, 127, 128, 131, 134, 144.3, 146.1, 147.9 (12C, Ar)	418M^+
4d	771 (C–Cl), 1333 (C–N), 1757 (CO cyclic), 2918 (CH–Cl)	4.53 (d, 1H, J = 5.10 Hz, CH–Cl), 5.26 (d, 1H, J = 5.10 Hz, N–CH), 5.92 (s, 1H, NH), 6.81–7.62 (m, 8H, Ar)	55.8 (CH–Cl), 61.4 (N–CH), 165.2 (CO cyclic), 115, 117.9, 121.7, 124.7, 126.6, 128.9, 130, 133, 135, 141.3, 145.9, 149.1 (12C, Ar)	418M^+
4e	578 (C–Br) 1315 (C–N), 1741 (CO cyclic), 2892 (CH–Cl)	4.62 (d, 1H, J = 5.15 Hz, CH–Cl), 5.42 (d, 1H, J = 5.15 Hz, N–CH), 5.87 (s, 1H, NH), 7.35–7.95 (m, 8H, Ar)	47.1 (CH–Cl), 59.1 (N–CH), 161.3 (CO cyclic),112, 113, 117.9, 120, 122.1, 125.6, 127.4, 128.8, 135, 139, 143, 147.9 (12C, Ar)	463M^+
4f	571 (C–Br), 1318 (C–N), 1748 (CO cyclic), 2896 (CH–Cl)	4.65 (d, 1H, J = 5.15 Hz, CH–Cl), 5.37 (d, 1H, J = 5.15 Hz, N–CH), 5.92 (s, 1H, NH), 7.31–7.92 (m, 8H, Ar)	48.7 (CH–Cl), 59.9 (N–CH), 165.3 (CO cyclic), 109, 114, 118.9, 120.7, 122.7, 125.6, 128.4, 129.8, 132.2, 139.2, 142.6, 147.1 (12C, Ar)	463M^+
4g	565 (C–Br), 1324 (C–N), 1755 (CO cyclic), 2884 (CH–Cl)	4.64 (d, 1H, J = 5.15 Hz, CH–Cl), 5.15 (d, 1H, J = 5.15 Hz, N–CH), 5.87 (s, 1H, NH), 7.27–7.84 (m, 8H, Ar)	47.7 (CH–Cl), 58.1 (N–CH), 162.5 (CO cyclic), 111, 115.4, 118.9, 120, 121.7, 124, 127.2, 130, 131, 140, 142.1, 147.9 (12C, Ar)	463M^+
4h	868 (C–NO), 1351 (C–N), 1538 (NO_2), 1741 (CO cyclic), 2921 (CH–Cl)	4.38 (d, 1H, J = 5.25 Hz, CH–Cl), 5.43 (d, 1H, J = 5.25 Hz, N–CH), 5.91 (s, 1H, NH), 7.1–7.71 (m, 8H, Ar)	51 (CH–Cl), 68.8 (N–CH), 161 (CO cyclic), 112, 118, 122, 124, 125, 126, 128, 132.8, 136.1, 144.3, 145.9, 147.9 (12C, Ar)	429M^+
4i	862 (C–NO), 1357 (C–N), 1542 (NO_2), 1749 (CO cyclic), 2914 (CH–Cl)	4.39 (d, 1H, J = 5.22 Hz, CH–Cl), 5.42 (d, 1H, J = 5.22 Hz, N–CH), 5.97 (s, 1H, NH), 7.16–7.79 (m, 8H, Ar)	54 (CH–Cl), 63.8 (N–CH), 167 (CO cyclic), 115, 118.9, 122.7, 124.8, 125.9, 126.8, 128.4, 132.2, 136.9, 142.3, 146.9, 147.1 (12C, Ar)	429M^+

(*continued on next page*)

Table 1 (*continued*)

Comp.	IR (cm^{-1})	^{1}H NMR	^{13}C NMR	FAB mass
4j	869 (C–NO), 1354 (C–N), 1542 (NO_2), 1745 (CO cyclic), 2918 (CH–Cl)	4.31 (d, 1H, J = 5.22 Hz, CH–Cl), 5.54 (d, 1H, J = 5.22 Hz, N–CH), 5.94 (s, 1H, NH), 7.05–7.71 (m, 8H, Ar)	54.6 (CH–Cl), 64.8 (N–CH), 163 (CO cyclic), 116.4, 117.4, 122, 123.8, 124.9, 125.2, 127.4, 130.8, 132.9, 145.7, 148.5, 151.1 (12C, Ar)	429M^+
4k	1163 (C–O), 1326 (N–C), 1736 (CO cyclic), 2891 (CH–Cl)	3.64 (s, 3H, OCH_3), 4.41 (d, 1H, J = 5.10 Hz, CH–Cl), 5.39 (d, 1H, J = 5.10 Hz, N–CH), 5.76 (s, 1H, NH), 7.26–7.92 (m, 8H, Ar)	49.1 (CH–Cl), 54 (OCH_3), 64.4 (N–CH), 162.5 (CO cyclic), 117, 122.4, 123, 124.8, 126.9, 127, 128.7, 129, 132, 141, 148, 159 (12C, Ar)	414M^+
4l	1168 (C–O), 1322 (N–C), 1728 (C═O cyclic), 2895 (CH–Cl)	3.59 (s, 3H, OCH_3), 4.49 (d, 1H, J = 5.10 Hz, CH–Cl), 5.29 (d, 1H, J = 5.10 Hz, N–CH), 5.70 (s, 1H, NH), 7.36–8.02 (m, 8H, Ar)	49.8 (CH–Cl), 54.8 (OCH_3), 62.4 (N–CH), 164.5 (CO, cyclic), 120, 123.4, 124, 125.8, 126, 127.5, 128, 129, 129.1, 145, 147, 159.6 (12C, Ar)	414M^+
4m	1163 (C–O), 1328 (N–C), 1738 (CO cyclic), 2885 (CH–Cl)	3.52 (s, 3H, OCH_3), 4.45 (d, 1H, J = 5.12 Hz, CH–Cl),5.39 (d, 1H, J = 5.12 Hz, N–CH), 5.77 (s, 1H, NH), 7.04–7.87 (m, 8H, Ar)	47.4 (CH–Cl), 54.1 (OCH_3), 63.1 (N–CH), 163.2 (CO cyclic), 117, 119.4, 122, 124.8, 126.8, 126.5, 127, 128.4, 135.9, 147, 147.8, 157.2 (12C, Ar)	414M^+
4n	1328 (C–N), 1740 (CO cyclic), 2889 (CH–Cl), 2924 (CH_3)	2.58 (s, 3H, CH_3), 4.53 (d, 1H, J = 5.0 Hz, CH–Cl), 5.42 (d, 1H, J = 5.0 Hz, N–CH), 5.68 (s, 1H, NH), 7.28–7.98 (m, 8H, Ar)	24.7 (CH_3), 51.7 (CH–Cl), 62.8 (N–CH), 164.8 (CO, cyclic), 117, 118.9, 120.7, 122.7, 124, 125.2, 128.3, 129.8, 132.2, 138, 146.9, 147.1 (12C, Ar)	398M^+
4o	1322 (C–N), 1746 (CO cyclic), 2894 (CH–Cl), 2927 (CH_3)	2.56 (s, 3H, CH_3), 4.57 (d, 1H, J = 5.0 Hz, CH–Cl), 5.34 (d, 1H, J = 5.0 Hz, N–CH), 5.62 (s, 1H, NH), 7.18–7.84 (m, 8H, Ar)	23.5 (CH_3), 51 (CH–Cl), 63.8 (N–CH), 163.8 (CO, cyclic), 117.5, 118.2, 122.7, 123.3, 124.5, 125.7, 127.3, 129.1, 131.2, 136, 146.1, 147.9 (12C, Ar)	398M^+
4p	1321 (C–N), 1746 (CO cyclic), 2876 (CH–Cl), 2913 (CH_3)	2.52 (s, 3H, CH_3),4.48 (d, 1H, J = 5.0 Hz, CH–Cl), 5.45 (d, 1H, J = 5.0 Hz, N–CH), 5.66 (s, 1H, NH), 7.21–8.09 (m, 8H, Ar)	23.4 (CH_3), 50.9 (CH–Cl), 62.2 (N–CH), 161.2 (CO cyclic), 116.4, 118.7, 120.1, 123.7, 125, 124.2, 127.3, 128.8, 132.8, 137.4, 145.4, 146.3 (12C, Ar)	398M^+
4q	1187 (C–O), 1355 (C–N), 1758 (CO cyclic), 2917 (CH–Cl), 3467 (OH)	4.20 (s, 1H, OH), 4.59 (d, 1H, J = 5.30 Hz, CH–Cl), 5.38 (d, 1H, J = 5.30 Hz, N–CH), 5.91 (s, 1H, NH), 7.09–8.1 (m, 8H, Ar)	53.2 (CH–Cl), 63.7 (N–CH), 166.4 (CO cyclic), 113.1, 121, 122, 122.3, 123.9, 124.1, 125, 127.1, 128.2, 132, 143.2, 145.6 (12C, Ar)	400M^+
4r	1182 (C–O), 1360 (C–N), 1762 (CO cyclic), 2923 (CH–Cl), 3469 (OH)	4.22 (s, 1H, OH), 4.57 (d, 1H, J = 5.27 Hz, CH–Cl), 5.41 (d, 1H, J = 5.27 Hz, N–CH), 5.93 (s, 1H, NH), 7.12–8.13 (m, 8H, Ar)	51.2 (CH–Cl), 64.7 (N–CH), 165.7 (CO cyclic), 113.9, 120.6, 124.2, 123.5, 124.6, 125, 125.5, 127.1, 128.7, 133, 141, 146 (12C, Ar)	400M^+
4s	1182 (C–O), 1359 (C–N), 1752 CO cyclic), 2919 (CH–Cl), 3459 (OH)	4.25 (s, 1H, OH), 4.57 (d, 1H, J = 5.27 Hz, CH–Cl), 5.39 (d, 1H, J = 5.27 Hz, N–CH), 5.99 (s, 1H, NH), 7.19–8.21 (m, 8H, Ar)	54 (CH–Cl), 63.2 (N–CH), 165.9 (CO, cyclic), 113.5, 121.3, 122, 122.6, 123.2, 124, 125.6, 127.8, 128.5, 132.6, 144, 146.1 (12C, Ar)	400M^+



Scheme for the synthesis of compounds 1, **2**, **3**(**a**–**s**), **4**(**a**–**s**).

Comp.	Ar	Comp.	Ar	Comp.	Ar
3a, 4a	C_6H_5	3h, 4h	4-$NO_2C_6H_4$	3o, 4o	2-$CH_3OC_6H_4$
3b, 4b	4-ClC_6H_4	3i, 4i	3-$NO_2C_6H_4$	3p, 4p	2-$CH_3OC_6H_4$
3c, 4c	3-ClC_6H_4	3j, 4j	2-$NO_2C_6H_4$	3q, 4q	4-HOC_6H_4
3d, 4d	2-ClC_6H_4	3k, 4k	4-$CH_3OC_6H_4$	3r, 4r	3-HOC_6H_4
3e, 4e	4-BrC_6H_4	3l, 4l	3-$CH_3OC_6H_4$	3s, 4s	2-HOC_6H_4
3f, 4f	3-BrC_6H_4	3m, 4m	2-$CH_3OC_6H_4$	–	–
3g, 4g	2-BrC_6H_4	3n, 4n	4-$CH_3C_6H_4$	–	–

2. Materials and methods

2.1. Experimental

Melting points were taken in open capillaries and are uncorrected. Progress of reaction was monitored by silica gel-G coated TLC plates using MeOH: $CHCl_3$ system (1:9). The spot

was visualized by exposing dry plate at iodine vapours chamber. IR spectra were recorded in KBr disc on a Schimadzu 8201 PC, FTIR spectrophotometer (ν_{max} in cm^{-1}) and ^1H NMR and ^{13}C NMR spectra were measured on a Brucker DRX-300 spectrometer in $CDCl_3$ at 300 MHz using TMS as an internal standard. All chemical shifts were reported on δ scales. The FAB mass spectra were recorded on a Jeol SX-102 mass spectrometer. Elemental analyses were performed on a Carlo Erba-1108 analyzer. Microwave irradiation carried out in open glass vessel. Modified microwave oven (800 W) was used for the synthesis of compounds. A thermocouple used to monitor the temperature inside the vessel of the microwave. The analytical data of all the compounds were highly satisfactory. For column chromatographic purification of the products, Merck silica Gel 60 (230–400 Mesh) was used. The reagent grade chemicals were purchased from the commercial sources and further purified before use (see Table 1).

2.2. Synthesis of the compound *1*

A solid supported mixture of 1,2,3-benzotriazole and 1-bromo-3-chloropropane (1:1 mole) was mixed thoroughly, takes in open glass vessel and subjected to the microwave irradiation at low power setting (25%, 200 W) for 4 min., then allowed to cool. The product was purified over a column chromatography. The purified product was recrystallized from methanol at room temperature to yield compound **1**.

2.3. Synthesis of the compound *2*

A solid supported mixture of compound **1** and urea (1:1 mole) was mixed thoroughly, takes in open glass vessel and subjected to the microwave irradiation at low power setting (25%, 200 W) for 3 min, then allowed to cool. The product was purified over a column chromatography. The purified product was

Table 2 Physical data of the compounds **1, 2, 3(a–s)** and **4(a–s)**.

Comp.	Mol. Formula	Mol. Wt.	Melting point (°C)	Yield (%)	Time (min)	Microanalytical data	
						Calculated	Found
1	$C_9H_{10}N_3Cl$	196	78–79	78	4.00	C55.25, H5.15, N21.47	C55.21, H5.13, N21.41
2	$C_{10}H_{13}N_5O$	219	70–73	85	3.00	C54.73, H5.97, N31.94	C54.70, H5.92, N31.89
3a	$C_{17}H_{17}N_5O$	307	80–83	75	3.30	C6.43, H5.53, N22.78	C66.41, H5.50, N22.75
3b	$C_{17}H_{16}N_5OCl$	341	80–82	80	3.45	C59.73, H4.71, N20.48	C59.61, H4.62, N20.38
3c	$C_{17}H_{16}N_5OCl$	341	76–79	82	3.30	C59.73, H4.71, N20.48	C59.59, H4.66, N20.41
3d	$C_{17}H_{16}N_5OCl$	341	80–82	81	4.00	C59.73, H4.71, N20.48	C59.61, H4.69, N20.39
3e	$C_{17}H_{16}N_5OBr$	386	80–81	83	3.00	C52.86, H4.17, N18.13	C52.82, H4.13, N18.07
3f	$C_{17}H_{16}N_5OBr$	386	78–80	85	3.15	C52.86, H4.17, N18.13	C52.72, H4.11, N18.02
3g	$C_{17}H_{16}N_5OBr$	386	79–81	78	2.45	C52.86, H4.17, N18.13	C52.81, H4.03, N18.11
3h	$C_{17}H_{16}N_6O_3$	352	96–98	79	3.10	C57.94, H4.57, N23.85	C57.81, H4.52, N23.63
3i	$C_{17}H_{16}N_6O_3$	352	92–93	78	3.20	C57.94, H4.57, N23.85	C57.85, H4.54, N23.61
3j	$C_{17}H_{16}N_6O_3$	352	93–94	82	2.50	C57.94, H4.57, N23.85	C57.91, H4.45, N23.57
3k	$C_{18}H_{19}N_5O_2$	337	76–78	79	3.45	C64.08, H5.67, N20.75	C63.96, H5.56, N20.65
3l	$C_{18}H_{19}N_5O_2$	337	73–75	83	3.30	C64.08, H5.67, N20.75	C63.98, H5.62, N20.63
3m	$C_{18}H_{19}N_5O_2$	337	72–74	80	4.00	C64.08, H5.67, N20.75	C63.91, H5.57, N20.61
3n	$C_{18}H_{19}N_5O$	321	80–82	76	4.15	C67.27, H5.95, N21.79	C67.15, H5.90, N21.73
3o	$C_{18}H_{19}N_5O$	321	77–78	78	4.10	C67.27, H5.95, N21.79	C,67.11, H5.85, N,21.77
3p	$C_{18}H_{19}N_5O$	321	78–80	76	4.00	C67.27, H5.95, N21.79	C,67.21, H5.89, N,21.73
3q	$C_{17}H_{17}N_5O_2$	323	85–86	79	3.20	C63.14, H5.29, N21.65	C63.07, H5.22, N21.53
3r	$C_{17}H_{17}N_5O_2$	323	82–83	79	3.30	C63.14, H5.29, N21.65	C63.11, H5.18, N21.58
3s	$C_{17}H_{17}N_5O_2$	323	83–84	78	3.20	C63.14, H5.29, N21.65	C63.09, H5.24, N21.58
4a	$C_{19}H_{18}N_5O_2Cl$	384	75–76	76	3.45	C59.45, H4.72, N18.24	C59.31, H4.62, N18.13
4b	$C_{19}H_{17}N_5O_2Cl_2$	418	79–80	77	3.30	C54.55, H4.14, N16.74	C54.48, H4.32, N16.63.
4c	$C_{19}H_{17}N_5O_2Cl_2$	418	77–79	84	3.20	C54.55, H4.14, N16.74	C54.42, H4.08, N16.56%
4d	$C_{19}H_{17}N_5O_2Cl_2$	418	76–78	80	3.15	C54.55, H4.14, N16.74	C54.48, H4.05, N16.78
4e	$C_{19}H_{17}N_5O_2BrCl$	463	74–75	82	3.30	C49.31, H3.70, N15.13	C49.19, H3.64, N15.05
4f	$C_{19}H_{17}N_5O_2BrCl$	463	72–73	81	3.10	C49.31, H3.70, N15.13	C49.22, H3.49, N15.03%
4g	$C_{19}H_{17}N_5O_2BrCl$	463	79–81	79	3.45	C49.31, H3.70, N15.13	C49.25, H3.43, N15.22
4h	$C_{19}H_{17}N_6O_4Cl$	429	78–79	78	3.30	C53.21, H3.99, N19.59	C53.14, H3.74, N19.49
4i	$C_{19}H_{17}N_6O_4Cl$	429	77–78	81	3.15	C53.21, H3.99, N19.59	C53.12, H3.82, N19.53
4j	$C_{19}H_{17}N_6O_4Cl$	429	79–81	80	3.00	C53.21, H3.99, N19.59	C53.18, H3.92, N19.51
4k	$C_{20}H_{20}N_5O_3Cl$	414	77–78	81	3.20	C58.04, H4.87, N16.92	C57.94, H4.72, N16.83
4l	$C_{20}H_{20}N_5O_3Cl$	414	79–81	76	3.30	C58.04, H4.87, N16.92	C57.91, H4.372, N16.83
4m	$C_{20}H_{20}N_5O_3Cl$	414	76–78	78	3.15	C58.04, H4.87, N16.92	C57.97, H4.32, N16.88
4n	$C_{20}H_{20}N_5O_2Cl$	398	75–77	75	3.45	C60.37, H5.06, N7.60	C60.21, H4.92, N17.53
4o	$C_{20}H_{20}N_5O_2Cl$	398	73–75	76	3.30	C60.37, H5.06, N7.60	C60.25, H4.95, N17.58
4p	$C_{20}H_{20}N_5O_2Cl$	398	72–73	79	3.30	C60.37, H5.06, N7.60	C60.27, H4.90, N17.49
4q	$C_{19}H_{18}N_5O_3Cl$	400	90–91	77	3.20	C57.07, H4.53, N17.51	C56.91, H4.42, N17.43
4r	$C_{19}H_{18}N_5O_3Cl$	400	86–87	80	3.15	C57.07, H4.53, N17.51	C56.99, H4.30, N17.35
4s	$C_{19}H_{18}N_5O_3Cl$	400	88–89	81	3.30	C57.07, H4.53, N17.51	C56.91, H4.36, N17.33

recrystallized from methanol at room temperature to yield compound **2**.

2.4. General methods for the synthesis of the compound 3(a–s)

A solid supported mixture of compound **2** and various substituted benzaldehydes (1:1 mole) was mixed thoroughly, takes in open glass vessel and subjected to the microwave irradiation at low power setting (25%, 200 W) for 2.45–4.15 min, then allowed to cool. The products were purified over a column chromatography. The purified products were recrystallized from methanol at room temperature to yield compound **3(a–s)**.

2.5. General methods for the synthesis of the compound 4(a–s)

A solid supported mixture of compound **3(a–s)** and $ClCH_2COCl$ in the presence of Et_3N (1:1:1 mole) was mixed thoroughly, takes in open glass vessel and subjected to the microwave irradiation at low power setting (25%, 200 W) for 3.10–3.45 min, then allowed to cool. The products were purified over a column chromatography. The purified products were recrystallized from methanol at room temperature to yield compound **4(a–s)**.

3. Results and discussion

N^1-[3-{(4-substitutedaryl-3-chloro-2-oxo-azetidine)-carbamyl} -propyl]-1,2,3-benzotriazoles **4(a–s)** were synthesized in four different steps. 1,2,3-benzotriazole on reaction with $Cl(CH_2)_3Br$ at room temperature afforded N^1-(3-chloropropyl)-1,2,3-benzotriazole, compound **1**. IR spectrum of compound **1** displayed absorption for (N–CH_2), (C–Cl), also indicated the disappearance of NH of 1,2,3-benzotriazole at (3442). The compound **1** on reaction with urea at room temperature yielded N^1-{3-(aminocarbamyl)-propyl}-1,2,3-benzotriazole, compound **2**. IR spectrum of compound **2** showed absorption for NH and CO while absorption of (C–Cl) has been disappeared. The ^{1}H NMR spectrum of compound **2** displayed a signal for (CH_2–N) and its ^{13}C NMR spectrum showed a signal for CO group. The compound **2** on further reaction with selected several substituted aromatic aldehydes produced N^1-{3-(substitutedarylidine carbamyl)-propyl-1,2,3-benzotriazole, compound **3(a–s)**. For the compound **3(a–s)** showed the characteristic absorption for Schiff bases (N═CH) in IR spectra and also displayed a characteristic signal in the ^{1}H and ^{13}C NMR spectra. In the ^{1}H NMR a broad signal of NH_2 has been disappeared. In the FAB–MS spectra of **3(a–s)** parent ion peaks found at appropriate value of their molecular weights. The compound **3(a–s)** on treatment with $ClCH_2COCl$ in the presence of Et_3N furnished final products compound **4(a–s)**. In the ^{13}C NMR spectra of compound **4(a–s)** showed three signals for (N–CH), (CH–Cl) and carbonyl group of β-lactam ring and ^{1}H NMR spectra displayed two doublet for (N–CH) and (CH–Cl). In the IR, ^{1}H and ^{13}C NMR spectra of compound **4(a–s)** signal for N═CH have been disappeared. Reaction time and yield of the compounds were given in Table 2.

4. Conclusion

In conclusion, microwave method is better for rapid synthesis for all above compounds and found high yield and purity. Reaction time and solvents use is very low.

Acknowledgement

The authors are thankful to SAIF, Indian institute of Technology New Delhi (India) and Central Drugs Research Institute, Lucknow (India) for providing spectral and analytical data of the compounds. We are also thankful to Head, Department of Chemistry Dr. H.S. Gour, University (A Central University), Sagar, M.P. (India) for giving the facilities to carryout the work.

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