Synthesis of New Tetrahydro-1,5-benzodiazepin-3-yl-2-phenylacetamides *via* Isocyanide-Based Multicomponent Reactions

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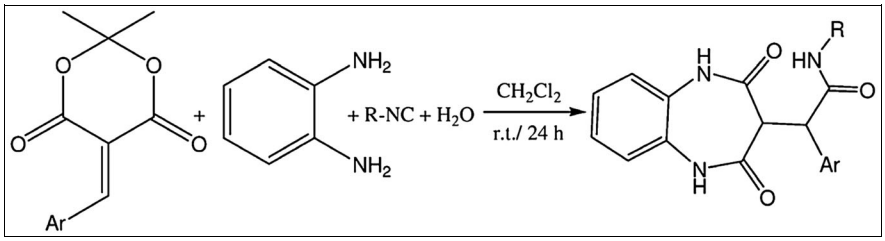

New tetrahydro-1*H*-1,5-benzodiazepin-2-phenylacetamides were synthesized in good yields by a four-component reaction of benzylidene Meldrum's acid, benzene-1,2-diamines, isocyanides, and water in CH_2Cl_2 at room temperature.

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INTRODUCTION

Benzodiazepine derivatives have been known to display a wide range of pharmacological activities as anticonvulsant, antianxiety, analgesic, sedative, antidepressive, hypnotic agents [1], as well as anti-inflammatory agents [2]. Among the 1,5-benzodiazepines, the 1,5-benzodiazepine-2-ones such as telenzepine [3], triflubazam [4], and clobazam [5] (Fig. 1), are clinically used as anxiolytic or antisecretory agents. Further, compound **I** have been investigated as cholecystokinin-B receptor antagonist [6] (Fig. 1). Two recently published patents indicate that 2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepine derivatives carrying carboxamide substituents are potentially important as a therapeutic and prophylactic agent for diabetes, diabetic nephropathy, or glomerulosclerosis [7].

Multicomponent reactions (MCRs) have been frequently used by synthetic chemists as a facile means to generate molecular diversity from bifunctional substrates that react sequentially in an intramolecular fashion [8,9]. In recent years, isocyanide-based multicomponent condensation reactions (IMCRs) by virtue of their synthetic potential, their inherent atom efficiency, convergent nature, ease of implementation, and the generation of molecular diversity, have attracted much attention because of the advantages that they offer to the field of combinatorial chemistry [10,11]. Similarly, IMCRs have emerged as valuable tools for the preparation of structurally diverse chemical libraries of drug-like heterocyclic compounds [12–16]. In this context, benzodiazepine derivatives show interesting features that make them attractive for use in IMCRs [17–23].

Because of the biological activity of 2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepine [7], and our interest in synthesis of heterocyclic compounds [24–28], herein, we report an efficient isocyanide-based four-component method for the preparation of new tetrahydro-1*H*-1,5-benzodiazepin-2-phenylacetamides. These compounds have closely related ring systems such as triflubazam, clobazam, and 1,5-benzodiazepines, which have a broad spectrum of biological activities.

RESULTS AND DISCUSSION

We found that a mixture of benzylidene Meldrum's acid **1**, benzene-1,2-diamines **2,** isocyanides **3,** and water in CH_2Cl_2 at room temperature, afforded corresponding 2,4-dioxo-2,x3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl-2-phenylacetamides **4** in good yields for 24 h. The results are summarized in Table 1.

The good yield and purity of products and simplicity of the present procedure makes it an interesting, convenient, and acceptable one-pot method for the preparation of tetrahydro-1*H*-1,5-benzodiazepin-2-phenylacetamides. In addition, the workup of these very clean reactions involves only a filtration and simple washing step with EtOH.

Because of the importance of ferrocenyl heterocyclic compounds [29,30], we used ferrocenecarboxaldehyde **5** in the reaction. This made it possible to synthesize new ferrocenyl benzodiazepine **6** (Scheme 1).

We have not established an exact mechanism for the formation of products **4**; however, a reasonable possibility is shown in Scheme 2. The formation of product **4** can be rationalized by initial formation of 1,5-benzodiazepine **7** *via* condensation of **1** and **2**. Intermediate **8** was produced by Michael-type addition reaction of an isocyanide **3** to **7**, followed by nucleophilic attack of a H_2O molecule on the nitrilium moiety to produce compound **9**. Finally, tautomerization

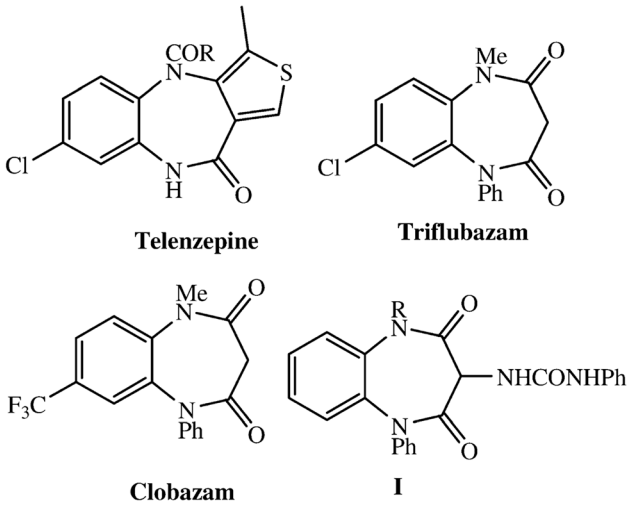


Figure 1. Important biologically active 1,5-benzodiazepines.

of **9** produces the tetrahydro-1*H*-1,5-benzodiazepin-2-phenylacetamides **4** (Scheme 2) [23]. Compounds 4 and 6 are stable solids whose structures were established by IR, ^{1}H NMR spectroscopy, and elemental analysis.

In conclusion, we have described an efficient four-component method for the synthesis of new tetrahydro-1,5-benzodiazepin-2-phenylacetamides using readily available starting materials. The products are of potential synthetic and pharmacological interest. Prominent among the advantages of this new method are operational simplicity, good yields, and easy work-up procedures employed.

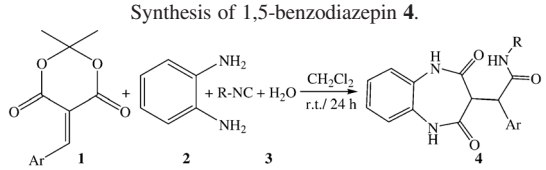
EXPERIMENTAL

Melting points were measured on an Electrothermal 9100 apparatus. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionization potential of 70 eV. ^{1}H NMR spectra were recorded on a BRUKER DRX-300 AVANCE spectrometer at 300.13. IR spectra were recorded using a Shimadzu IR-470 apparatus. Elemental analyses were performed using a Heracus CHN-O-Rapid analyzer.

Because of the very low solubility of products **4** and **6**, we were unable to obtain the ^{13}C-NMR spectrum.

Table 1

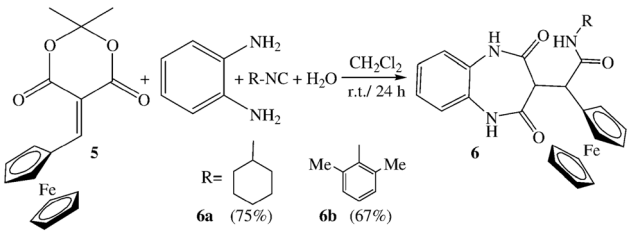
Synthesis of 1,5-benzodiazepin **4**.



Product **4**	Ar	R′	Yield (%)[a]
a	C_6H_5	Cyclohexyl	82
b	4-Cl-C_6H_4	Cyclohexyl	80
c	2-Cl-C_6H_4	Cyclohexyl	76
d	4-Me-C_6H_4	Cyclohexyl	79
e	4-Br-C_6H_4	Cyclohexyl	77
f	3-Br-C_6H_4	Cyclohexyl	73
g	3-NO_2-C_6H_4	Cyclohexyl	81
h	3-MeO-C_6H_4	Cyclohexyl	82
i	C_6H_5	2,6-$(Me)_2C_6H_3$	77
j	4-Cl-C_6H_4	2,6-$(Me)_2C_6H_3$	74
k	3-MeO-C_6H_4	2,6-$(Me)_2C_6H_3$	79

[a]Isolated yields.

Scheme 1

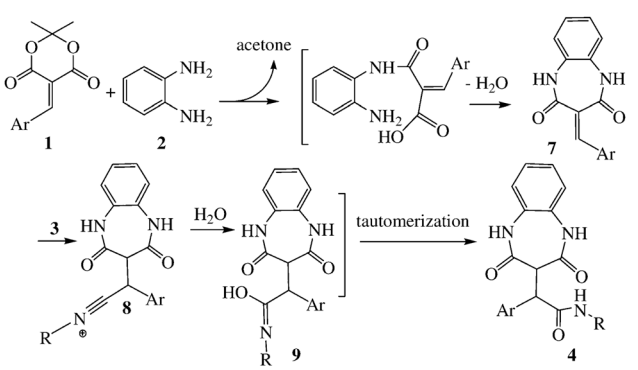


Typical procedure for the preparation of tetrahydro-1*H*-1,5-benzodiazepin-2-phenylacetamides (4). A mixture of benzylidene Meldrum's acid (1 mmol), benzene-1,2-diamines (1 mmol), isocyanides (1 mmol) and H_2O (0.5 mL) in CH_2Cl_2 (4 mL) was stirred for 24 h at room temperature. After completion of the reaction (TLC; ethyl acetate/*n*-hexane, 1/1), the reaction mixture was filtered and the precipitate washed with EtOH (5 mL) to afford the pure product.

***N*-cyclohexyl-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl)-2-phenylacetamide (4a).** White powder (82%); mp >260°C. ir (KBr) (ν_{max}/cm^{-1}): 3331, 3283, 3065, 1700, 1649, 1606. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H 0.99–1.74 (10H, m, 5CH_2 of cyclohexyl), 3.35 (1H, bs, CH-N, overlap with solvent), 3.74 (1H, d, J = 10.4 Hz, CH), 4.33 (1H, d, J = 10.4 Hz, CH), 7.11–7.39 (9H, m, Ar-H), 8.03 (1H, bs, NH-cyclohexyl), 10.29 (1H, s, NH), 10.49(1H, s, NH). MS (EI, 70 eV) *m/z*: 391 (M^+). Anal. Calcd for $C_{23}H_{25}N_3O_3$: C, 70.57; H, 6.44; N, 10.73. Found: C, 70.48; H, 6.38; N, 10.65.

2-(4-Chlorophenyl)-*N*-cyclohexyl-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl) acetamide (4b). White powder (80%); mp >260°C. ir (KBr) (ν_{max}/cm^{-1}): 3319, 3216, 3074, 1699, 1646, 1602. ^{1}H NMR (300 MHz, DMSO-d_6): δ_H 0.98–1.65 (10H, m, 5CH_2 of cyclohexyl), 3.36 (1H, bs, CH-N, overlap with solvent), 3.71 (1H, d, J = 11.3 Hz, CH), 4.35 (1H, d, J = 11.3 Hz, CH), 7.10–7.42 (8H, m, Ar-H), 8.08 (1H, d, J = 7.1 Hz, NH-cyclohexyl), 10.34 (1H, s, NH), 10.53 (1H, s, NH). MS

Scheme 2



(EI, 70 eV) *m/z*: 425 (M^+). Anal. Calcd for $C_{23}H_{24}ClN_3O_3$: C, 64.86; H, 5.68; N, 9.87. Found: C, 64.74; H, 5.59; N, 9.96.

2-(2-Chlorophenyl)-*N*-cyclohexyl-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl) acetamide (4c). White powder (76%); mp >260°C. ir (KBr) (ν_{max}/cm^{-1}): 3346, 3265, 3080, 1701, 1651, 1605. ^1H NMR (300 MHz, DMSO-d_6): δ_H 1.08–1.69 (10H, m, $5CH_2$ of cyclohexyl), 3.47 (1H, bs, CH-N, overlap with solvent), 3.95 (1H, d, *J* = 11.2 Hz, CH), 4.72 (1H, d, *J* = 11.2 Hz, CH), 7.14–7.37 (8H, m, Ar-H), 7.57 (1H, d, *J* = 8.1 Hz, NH-cyclohexyl), 10.31 (1H, s, NH), 10.62 (1H, s, NH). MS (EI, 70 eV) *m/z*: 425 (M^+). Anal. Calcd for $C_{23}H_{24}ClN_3O_3$: C, 64.86; H, 5.68; N, 9.87. Found: C, 64.94; H, 5.48; N, 9.80.

***N*-cyclohexyl-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl)-2-(4-methylphenyl) acetamide (4d).** White powder (79%); mp >260°C. ir (KBr) (ν_{max}/cm^{-1}): 3324, 3214, 3075, 1699, 1647, 1603. ^1H NMR (300 MHz, DMSO-d_6): δ_H 0.94–1.65 (10H, m, $5CH_2$ of cyclohexyl), 2.20 (3H, s, CH_3), 3.36 (1H, bs, CH-N, overlap with solvent), 3.69 (1H, d, *J* = 11.3 Hz, CH), 4.28 (1H, d, *J* = 11.2 Hz, CH), 6.97–7.27 (8H, m, Ar-H), 7.99 (1H, d, *J* = 7.8 Hz, NH-cyclohexyl), 10.26 (1H, s, NH), 10.48 (1H, s, NH). MS (EI, 70 eV) *m/z*: 405 (M^+). Anal. Calcd for $C_{24}H_{27}N_3O_3$: C, 71.09; H, 6.71; N, 10.36. Found: C, 70.99; H, 6.79; N, 10.45.

2-(4-Bromophenyl)-*N*-cyclohexyl-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl)acetamide (4e). White powder (77%); mp >270°C. ir (KBr) (ν_{max}/cm^{-1}): 3315, 3214, 3071, 1699, 1647, 1603. ^1H NMR (300 MHz, DMSO-d_6): δ_H 0.99–1.66 (10H, m, $5CH_2$ of cyclohexyl), 3.33 (1H, bs, CH-N, overlap with solvent), 3.73 (1H, d, *J* = 8.8 Hz, CH), 4.35 (1H, d, *J* = 8.8 Hz, CH), 6.95–7.49 (8H, m, Ar-H), 8.03 (1H, bs, NH-cyclohexyl), 10.28 (1H, s, NH), 10.47 (1H, s, NH). MS (EI, 70 eV) *m/z*: 471 (M^+), 469 (M^+). Anal. Calcd for $C_{23}H_{24}BrN_3O_3$: C, 58.73; H, 5.14; N, 8.93. Found: C, 58.81; H, 5.21; N, 8.82.

2-(3-Bromophenyl)-*N*-cyclohexyl-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl) acetamide (4f). White powder (73%); mp >270°C. ir (KBr) (ν_{max}/cm^{-1}): 3317, 3185, 3069, 1700, 1648, 1601. ^1H NMR (300 MHz, DMSO-d_6): δ_H 1.14–1.65 (10H, m, $5CH_2$ of cyclohexyl), 3.38 (1H, bs, CH-N, overlap with solvent), 3.71 (1H, d, *J* = 9.6 Hz, CH), 4.32 (1H, d, *J* = 9.6 Hz, CH), 7.20–7.48 (8H, m, Ar-H), 8.10 (1H, bs, NH-cyclohexyl), 10.37 (1H, s, NH), 10.55 (1H, s, NH). MS (EI, 70 eV) *m/z*: 471 (M^+), 469 (M^+). Anal. Calcd for $C_{23}H_{24}BrN_3O_3$: C, 58.73; H, 5.14; N, 8.93. Found: C, 58.60; H, 5.07; N, 8.81.

***N*-cyclohexyl-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl)-2-(3-nitrophenyl) acetamide (4g).** White powder (81%); mp >260°C. ir (KBr) (ν_{max}/cm^{-1}): 3316, 3216, 3069 (NH), 1700, 1646, 1605. ^1H NMR (300 MHz, DMSO-d_6): δ_H 0.94–1.76 (10H, m, $5CH_2$ of cyclohexyl), 3.36 (1H, bs, CH-N, overlap with solvent), 3.79 (1H, d, *J* = 11.3 Hz, CH), 4.52 (1H, d, *J* = 11.3 Hz, CH), 7.08-8.21 (7H, m, Ar-H), 8.05 (1H, d, *J* = 8 Hz, NH-cyclohexyl), 8.18–8.21 (2H, m, Ar-H), 10.38 (1H s, NH), 10.60 (1H, s, NH). MS (EI, 70 eV) *m/z*: 436 (M^+). Anal. Calcd for $C_{23}H_{24}N_4O_5$: C, 63.29; H, 5.54; N, 12.84. Found: C, 63.36; H, 5.62; N, 12.75.

***N*-cyclohexyl-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl)-2-(3-methoxyphenyl) acetamide (4h).** White powder (82%); mp >260°C. ir (KBr) (ν_{max}/cm^{-1}): 3361, 3260, 3060, 1690, 1677, 1605. ^1H NMR (300 MHz, DMSO-d_6): δ_H 1.15–1.65 (10H, m, $5CH_2$ of cyclohexyl), 3.38 (1H, bs, CH-N, overlap with solvent), 3.67 (4H, bs, OCH_3 and CH), 4.29 (1H, bs, CH), 6.77–7.21 (8H, m, Ar-H), 8.03 (1H, bs, NH-cyclohexyl), 10.30 (1H, s, NH), 10.49 (1H, s, NH). MS (EI, 70 eV) *m/z*: 421 (M^+). Anal. Calcd for $C_{24}H_{27}N_3O_4$: C, 68.39; H, 6.46; N, 9.97. Found: C, 68.28; H, 6.38; N, 10.09.

***N*-(2,6-dimethylphenyl)-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl)-2-phenylacetamide (4i).** White powder (77%); mp >260°C. ir (KBr) (ν_{max}/cm^{-1}): 3450, 3256, 3064, 1702, 1656, 1601. ^1H NMR (300 MHz, DMSO-d_6): δ_H 1.88 (6H, s, $2CH_3$), 3.84 (1H, d, *J* = 10.8 Hz, CH), 4.64 (1H, d, *J* = 10.8 Hz, CH) 6.96–7.55 (12H, m, Ar-H). 9.40 (1H, s, NH), 10.16 (1H, s, NH), 10.63 (1H, s, NH). MS (EI, 70 eV) *m/z*: 413 (M^+). Anal. Calcd for $C_{25}H_{23}N_3O_3$: C, 72.62; H, 5.61; N, 10.16. Found: C, 72.79; H, 5.55; N, 10.23.

2-(4-Chlorophenyl)-*N*-(2,6-dimethylphenyl)-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl)acetamide (4j). White powder (73%); mp >260°C. ir (KBr) (ν_{max}/cm^{-1}): 3450, 3300, 3290, 1699, 1662, 1600. ^1H NMR (300 MHz, DMSO-d_6): δ_H 1.94 (6H, s, $2CH_3$), 3.81 (1H, d, *J* = 12.9 Hz, CH), 4.65 (1H, d, *J* = 12.9 Hz, CH) 6.97–7.54 (11H, m, Ar-H). 9.65 (1H, s, NH), 10.39 (1H, s, NH), 10.67 (1H, s, NH). MS (EI, 70 eV) *m/z*: 447 (M^+). Anal. Calcd for $C_{25}H_{22}ClN_3O_3$: C, 67.04; H, 4.95; N, 9.38. Found: C, 67.14; H, 4.87; N, 9.31.

2-(3-Methoxyphenyl)-*N*-(2,6-dimethylphenyl)-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl)acetamide (4k). White powder (79%); mp >260°C. ir (KBr) (ν_{max}/cm^{-1}): 3291, 3228, 3078, 1700, 1656. ^1H NMR (300 MHz, DMSO-d_6): δ_H 1.92 (6H, s, $2CH_3$), 3.77 (3H, s, OCH_3), 3.85 (1H, d, *J* = 11.2 Hz, CH), 4.62 (1H, d, *J* = 11.2 Hz, CH), 6.76–7.20 (11H, m, Ar-H), 9.40 (1H, s, NH), 10.17 (1H, s, NH), 10.42 (1H, s, NH). MS (EI, 70 eV) *m/z*: 443 (M^+). Anal. Calcd for $C_{26}H_{25}N_3O_4$: C, 70.41; H, 5.68; N, 9.47. Found: C, 70.30; H, 5.61; N, 9.55.

***N*-cyclohexyl-2-ferocenyl-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl)acetamide (6a).** Light yellow (75%); mp >260°C. ir (KBr) (ν_{max}/cm^{-1}): 3460, 3300, 3074, 1700, 1655, 1603. ^1H NMR (300 MHz, DMSO-d_6): δ_H 1.19–1.83 (10H, m, $5CH_2$ of cyclohexyl), 3.56 (1H, bs, CH-N), 3.94–4.18 (11H, m, CH_{fer} and 2CH), 7.08–7.18 (4H, m, Ar-H). 8.10 (1H, d, *J* = 7.6 Hz, NH), 10.38 (1H, s, NH), 10.40 (1H, s, NH). MS (EI, 70 eV) *m/z*: 499 (M^+). Anal. Calcd for $C_{27}H_{29}FeN_3O_3$: C, 64.94; H, 5.85; N, 8.41. Found: C, 64.81; H, 5.76; N, 8.49.

***N*-(2,6-dimethylphenyl)-2-ferocenyl-2-(2,4-dioxo-2,3,4,5-tetrahydro-1*H*-1,5-benzodiazepin-3-yl)acetamide (6b).** White powder (67%); mp 228–235°C. ir (KBr) (ν_{max}/cm^{-1}): 3450, 3300, 3261, 1680, 1662, 1600. ^1H NMR (300 MHz, DMSO-d_6): δ_H 2.19 (6H, s, $2CH_3$), 4.22–4.80 (11H, m, CH_{fer} and 2CH), 6.53–7.31 (7H, m, Ar-H). 9.09 (1H, s, NH), 9.20 (1H, s, NH), 9.65 (1H, s, NH). MS (EI, 70 eV) *m/z*: 521 (M^+). Anal. Calcd for $C_{29}H_{27}FeN_3O_3$: C, 66.80; H, 5.22; N, 8.06. Found: C, 66.89; H, 5.28; N, 7.99.

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