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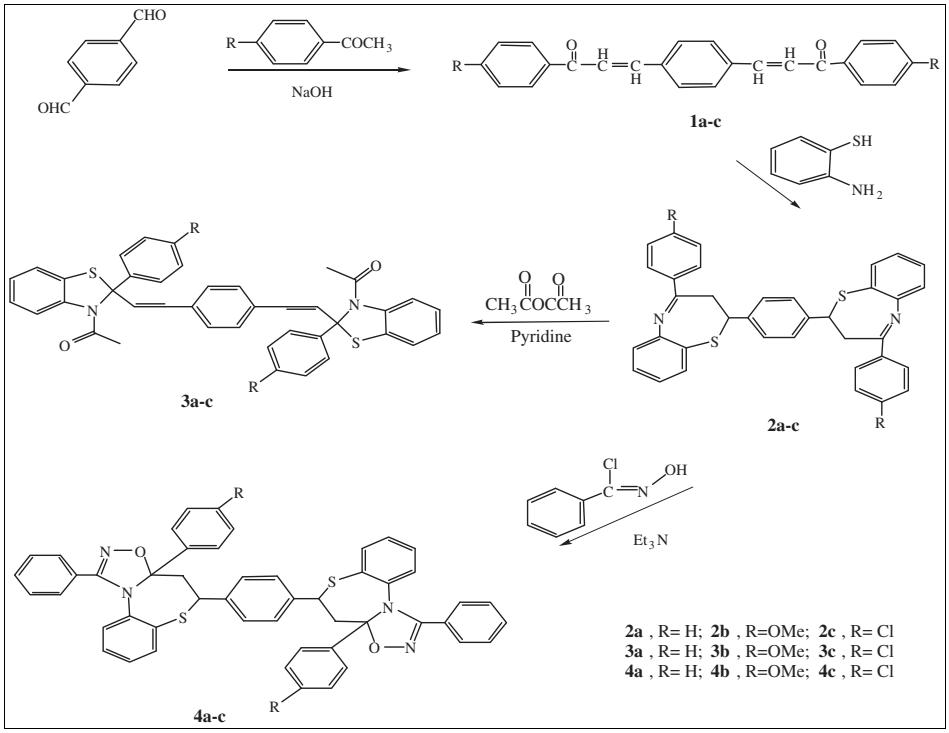

Some 1,4-phenylene-bis[1,2,4]oxadiazolo-[5,4-*d*][1,5]benzothiazepine derivatives (**4a–c**) were synthesized by 1,3-dipolar cycloaddition reaction of benzohydroximinoyl chloride with 1,4-phenylene-bis(4-aryl)-2,3-dihydro[1,5]benzothiazepine (**2a–c**); meanwhile, compounds **2a–c** also occurred ring contraction under acylating condition to obtain bis[2-aryl-2′-(β-1,4-phenylenevinyl)-3-acetyl]-2,3-dihydro[1,5]benzothiazoles (**3a–c**). The structures of some novel compounds were confirmed by IR, ^{1}H-NMR, elemental, and X-ray crystallographic analysis.

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INTRODUCTION

The synthesis of benzothiazepine derivatives had attracted considerable attention because of their broad spectrum of biological activities, such as cardiovascular modulator, coronary vasodilators, ACE inhibitors, anti-HIV, antihypertensive, antidepressant, antibacterial, and anticancer activity [1–3]. Diltiazem and Clentiazem were widely used as calcium channel blockers. Recently, progress had been made into fix an additional heterocycle on the heptatomic nucleus of 1,5-benzothiazepine for the preparation of tricyclic compounds that were found to be very potent nonnucleoside reverse transcriptase inhibitors [4]. The presence of the conformational preferences of the seven-membered ring was possibly correlated with biological activity and the fusion of a heterocyclic nucleus to the thiazepine system could induce an increase of the ring inversion barrier and consequently modify the activity profile [5,6].

1,2,4-Oxadiazole ring was also a major five-membered heterocyclic ring, which served as the core component of many substances that displayed a wide range of biological activities including apoptosis inducers, anticancer [7], anti-HIV [8], and selective cathepsin inhibitors [9]. In addition, many thiazoles had emerged as active pharmaceutical ingredients in several drugs because of their potential anti-inflammatory, antitumor [10], antihyperlipidemic, and antihypertensive [11] properties. Several methods for the synthesis of thiazoles had been developed, in particular for the 2-substituted benzothiazole. Most common synthetic methods used for their preparation were based on the condensation of 2-aminothiophenol with substituted carboxylic

acids, acyl chlorides, aldehydes, and nitriles [12]. Literature had reported a simple and convenient method of the synthesis of 2,3-dihydrobenzothiazoles by ring contraction of 2,3-dihydro[1,5]benzothiazepines under acylating reaction conditions [13]. But the study of the 1,3-dipolar cycloaddition and ring contraction of bis-2,3-dihydro[1,5] benzothiazepines were scarcely.

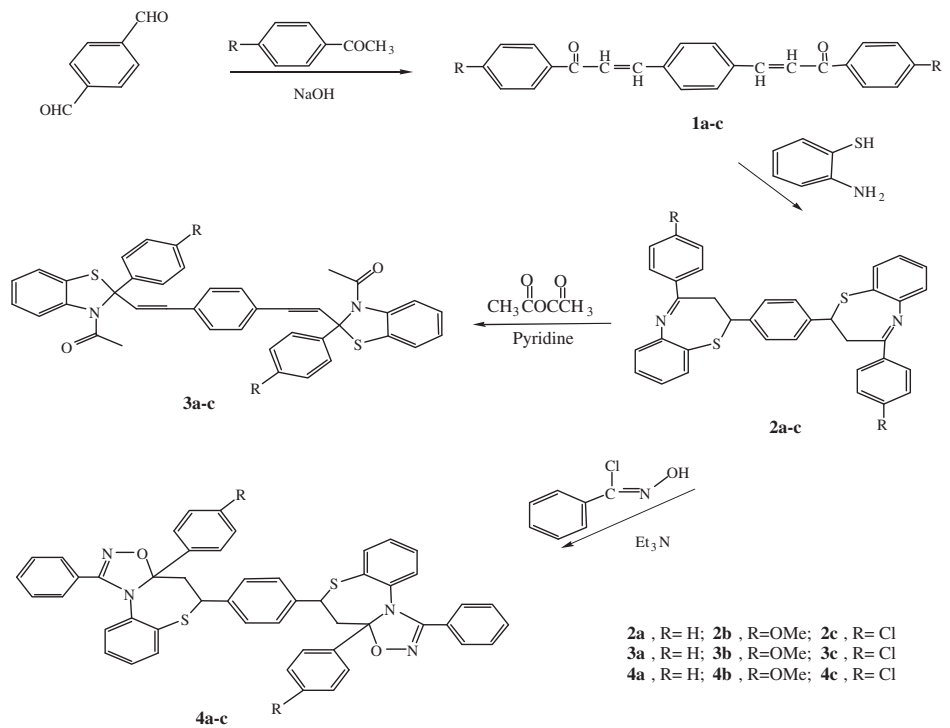
Inspired by the result of a research that dimers had shown more excellent activities than a monomer [14], the present investigation deals with the synthesis of novel 1,4-phenylene-bis(4-substituted phenyl)benzothiazepines and study their 1,3-dipolar cycloaddition and ring contraction as outlined in Scheme 1. These compounds might have useful biological and therapeutic activities. The crystal structure of **4c** determined by single-crystal X-ray diffraction was reported.

RESULTS AND DISCUSSION

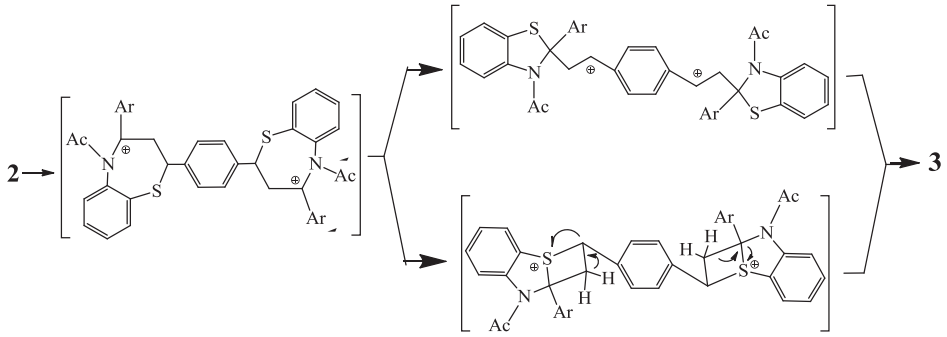
A series of novel bis-benzodiazepine derivatives containing 1,2,4-oxadiazole moiety was synthesized using terephthalic aldehyde as a starting material. The intermediate compounds, bis-benzodiazepine derivatives **2a–c**, were synthesized by reacting of α,β-unsaturated ketone **1a–c** with double equivalent *o*-aminobenzenethiol in ethanol using acetic acid as catalyst. Finally, the compounds **2a–c** underwent 1,3-dipolar cycloaddition with benzohydroximinoyl chloride in the presence of Et_3N leading to the formation of cycloadducts **4a–c**, yet underwent ring contraction with acetic anhydride leading to the formation of ring contraction products **3a–c**. The mechanism of ring contraction of simple 2,4-diaryl-2,3-dihydro-1,5-benzothiazepines had been proposed by Lévai, A., previously [15]. According to their interpretation, conversion of 2,4-diaryl-2,3-dihydro-1,5-benzothiazepines (**2**) into 2-styrylbenzothiazole (**3**) may start with acetylation of the nitrogen atom of benzothiazepine, followed either by heterolytic S—(C-2) bond scission and subsequent nucleophilic attack of the sulfur atom at C-4 affording an ally1 cation, or by a nucleophilic attack of sulfur as mentioned earlier giving rise to a cyclic sulfonium salt, deprotonation of which furnishes **3** (Scheme 2). The structures of all the synthesized compounds were established on the basis of their spectroscopic data; there was not ^{1}H-NMR data of the compounds **4a–c** because of their poor solubility.

In IR spectra, the synthesized final compounds **3a–c** showed a strong stretching vibration at 1672–1673 cm^{-1} because of the presence of C═O; compounds **4a–c** showed a strong stretching vibration at 1605–1608 cm^{-1} because of the presence of C═N. The ^{1}H-NMR spectrum revealed two distinct doublets at δ 6.86–6.90 and 6.51–6.54 ppm were attributed to CH═CH for compounds **3a–c**, respectively, and $J = 15.7$ Hz, which indicated the transconfiguration. The presence of a singlet at δ 2.09 ppm was attributed to the $COCH_3$. In MS spectra, molecular ions peaks of all target compounds were attained from EIMS. The molecular

Scheme 1. Synthetic process of the title compounds.



Scheme 2. The mechanism of ring contraction.



structure of compound **4c** was also confirmed by X-ray diffraction analysis, as shown in Figure 1. Detailed information about the crystal data and structure determination was summarized in Table 1. Selected interatomic distances and bond angles were tabulated in Table 2. Intermolecular interactions (Å) were listed in Table 3.

Although the compound **4c** had an abundance of conventional hydrogen-bond acceptors, it was a complete lack of donors. Thus, the packing features C—H group acted as donor in weak hydrogen-bond; however, the crystal was stabilized by weak offset face-to-face π···π interactions between cyclical 1 and cyclical 2, which was shown in Table 3, Figure 2. Other weak forces such as hydrophobic and dipole–dipole interactions also contributed to stabilization, but documenting them had proved to be more challenging than the study of H-bonding [16], the associated centroid–centroid distance between the benzene ring of Cg3|Cg3 was 3.6503 Å.

Suitable single crystals of the complex of dimension $0.67 \times 0.52 \times 0.28\,mm^3$, single-crystal diffraction data for **4c** were collected on a Bruker Smart Apex Duo diffractometer (Germany Bruker Company's Single Crystal Diffraction) at 296(2) K with Mo–Kα radiation (k = 0.71073 Å). Semiempirical absorption corrections were applied using SADABS program. All the structures were solved by direct methods using the SHELXS program of the SHELXTL package and refined by full-matrix least-squares methods on F^2 with SHELXL 97 [17]. Molecular structure was checked using PLATON [18]. 1,5-Benzothiazepine ring that was characterized by the endocyclic torsion angles adopts a boat-like conformation, The four atoms of C(17), C(25), N(1), and S(1) were closely coplanar; C(1), C(6), and C(30) were above the plane, with their deviations being −1.2236, −1.1852, and −0.6417 Å, respectively. The five-membered ring plane adopted an envelope conformation with atom C (17) deviating from the plane defined by C(8) N(1), N(2), and O(1) of 0.4993 Å. Cg(2) [center of gravity of the benzene ring (C1–C6)] was almost perpendicular with Cg(3) that dihedral angle was 91.0°, dihedral angle between oxadiazole ring Cg(1) and Cg(4) [center of gravity of the benzene ring (C18–C23)] was 91.0°, whereas with Cg(3) was 28.7°.

EXPERIMENTAL SECTION

All reagents were of commercial availability. Reactions were monitored by TLC. Melting points were measured on a mettler FP-5 capillary melting point apparatus and were uncorrected. Elemental analyses were conducted on a Perkin-Elmer 2400 elemental analyzer. The IR spectra were determined as potassium bromide pellet on a Bruker Equinox 55 FTIR spectrophotometer. Mass spectra were recorded on an Agilent 5975 apparatus (EI, 70 eV). X-ray crystal structure was obtained using R-AXIS SPIDER X-ray diffraction. Compounds **1a–c** [19,20] and benzenecarboximidoyl chloride [21] were synthesized according to the reported literatures.

General procedure for the synthesis of compounds 2a–c. A mixture of bischalcone (**1**) (5 mmol) and *o*-aminothiophenol (12 mmol) in anhydrous ethanol (50 mL) was heated under reflux for 8 h in the presence of acetic acid (1.0 mL). The mixture was cooled to RT, and then the solid product was separated by filtration and recrystallized from DMF/H_2O to give corresponding compounds **2a–c**.

1,4-Phenylene-bis(4-phenyl)-2,3-dihydro[1,5]benzothiazepine (2a). Pale yellow solid (70%); mp 199–200°C; FTIR ν 1610 (C═N), 1324 (C—N), 1241 (C—O—C) cm^{-1}; 8.09–7.16 (m, 22H, ArH), 4.98 (dd, 2H, H_{2x}, $J_{ax} = 9.2$ Hz, $J_{bx} = 2.0$ Hz), 3.33 (dd, 2H, H_{3b}, $J_{bx} = 2.0$ Hz, $J_{ab} = 4.5$ Hz), 3.06 (dd, 2H, H_{3a}, $J_{ax} = 9.2$ Hz, $J_{ab} = 4.5$ Hz); MS (ESI): *m/z* 553 ($M^+ + 1$); *Anal*. Calcd for $C_{36}H_{28}N_2S_2$: C, 78.22; H, 5.11; N, 5.07; S, 11.60; Found: C, 78.20; H, 5.11; N, 5.08; S, 11.61.

1,4-Phenylene-bis(4-chlorophenyl)-2,3-dihydro[1,5]benzothiazepine (2b). Pale white solid (75%); mp 224–225°C; FTIR ν 1606 (C═N), 1318 (C—N), 1244 (C—O—C) cm^{-1}; ^{1}H-NMR δ 7.97–7.15 (m, 20H, ArH), 4.95 (dd, 2H, H_{2x}, $J_{ax} = 9.6$ Hz, $J_{bx} = 2.6$ Hz), 3.24 (dd, 2H, H_{3b}, $J_{bx} = 2.6$ Hz, $J_{ab} = 8.5$ Hz), 3.04 (dd, 2H, H_{3a}, $J_{ax} = 9.6$ Hz, $J_{ab} = 8.5$ Hz); MS (ESI): *m/z* 621 ($M^+ + 1$); *Anal*. Calcd for $C_{36}H_{26}Cl_2N_2S_2$: C, 69.56; H, 4.22; Cl, 11.41; N, 4.51; S, 10.32; Found: C, 69.57; H, 4.21; Cl, 11.42; N, 4.50; S, 10.32.

1,4-Phenylene-bis(4-methoxyphenyl)-2,3-dihydro[1,5]benzothiazepine (2c). Pale yellow solid (72%); mp 226–228°C; FTIR ν 1597 (C═N), 1349 (C—N), 1261 (C—O—C) cm^{-1}; ^{1}H-NMR δ 8.02–7.16 (m, 20H, ArH), 4.97 (dd, 2H, H_{2x}, $J_{ax} = 9.1$ Hz, $J_{bx} = 2.6$ Hz), 3.83 (s, 6H, —OCH_3), 3.30 (dd, 2H, H_{3b}, $J_{bx} = 2.6$ Hz, $J_{ab} = 4.6$ Hz), 3.04 (dd, 2H, H_{3a}, $J_{ax} = 9.1$ Hz, $J_{ab} = 4.6$ Hz); MS (ESI): *m/z* 613 ($M^+ + 1$); *Anal*. Calcd for $C_{38}H_{32}N_2O_2S_2$: C, 74.48; H, 5.26; N, 4.57; O, 5.22; S, 10.47; Found: C, 74.47; H, 5.26; N, 4.56; O, 5.23; S, 10.47.

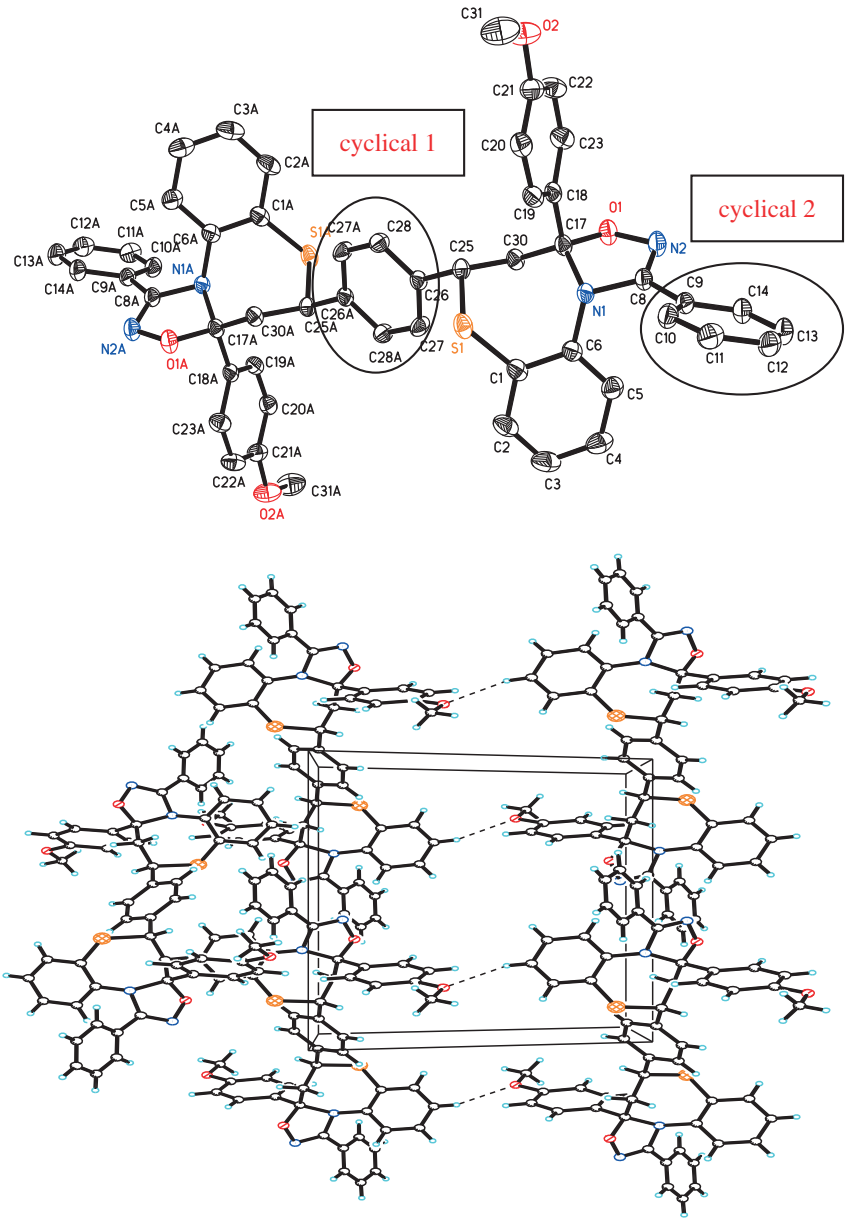


Figure 1. ORTEP representation and packing diagram of compound **4c**. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

General procedure for the synthesis of compounds 3a–c [16]. A mixture of 1,4-phenylene-bis(4-substituented phenyl) [1,5]benzothiazepine (**2**) (5 mmol), acetic anhydride (15 mL), and anhydrous pyridine (8 mL) was maintained at 80°C for 8–10 h (monitored by TLC) and then poured into water. The precipitate was filtered off, washed with water, and purified by silica gel column chromatography (ethylacetate/petroleum ether = 1:5, v/v) to afford the desired products **3a–c**.

Bis[2-phenyl-2′-(β-1,4-phenylenevinyl)-3-acetyl]-2,3-dihydro benzothiazole (3a). Pale yellow solid (74%); mp 119–120°C; FTIR ν 1672 (C═O), 1319 (C—N), 970 (CH═CH, trans) cm^{-1}; ^{1}H-NMR δ 7.04–7.62 (m, 22H, ArH), 6.86–6.91 (d, 2H, J = 15.7 Hz, benzothiazole ring—CH═), 6.51–6.56 (d, J = 15.7 Hz, 2H, benzene ring—CH═), 2.09 (s, 6H, $COCH_3$); MS (ESI): *m/z* 637 ($M^+ + 1$); *Anal*. Calcd for $C_{40}H_{32}N_2O_2S_2$: C, 75.44; H, 5.06; N, 4.40; O, 5.02; S, 10.07; Found: C, 75.43; H, 5.05; N, 4.42; O, 5.02; S, 10.07.

Bis[2-chlorophenyl-2′-(β-1,4-phenylenevinyl)-3-acetyl]-2,3-dihydrobenzothiazole (3b). Pale white solid (75%); mp 134–135°C; FTIR ν 1673 (C═N), 1320 (C—N), 970 (CH═CH, trans) cm^{-1}; ^{1}H-NMR δ 7.03–7.62 (m, 20H, ArH), 6.85–6.90 (d, J = 15.7 Hz, 2H, benzothiazole ring—CH═), 6.51–6.54 (d, J = 15.7 Hz, 2H, benzene ring—CH═), 2.09 (s, 6H, $COCH_3$); MS (ESI): *m/z* 705 ($M^+ + 1$); *Anal*. Calcd for $C_{40}H_{30}Cl_2N_2O_2S_2$: C, 68.08; H, 4.28; Cl, 10.05; N, 3.97; O, 4.53; S, 9.09; Found: C, 68.07; H, 4.28; Cl, 10.05; N, 3.98; O, 4.53; S, 9.09.

Bis[2-methoxyphenyl-2′-(β-1,4-phenylenevinyl)-3-acetyl]-2,3-dihydrobenzothiazole (3c). Pale yellow solid (68%); mp

Table 1

Crystal data and structure refinement of compound **4c**.

Formula	C_{52} H_{46} N_4 O_6 S_2
Crystal color	Colorless
Formula weight	887.05
Temperature (K)	296(2)
Crystal size (mm)	$0.67 \times 0.52 \times 0.28$
Crystal system	Monoclinic
Space group	P21/c
a(Å)	12.6667(7)
b(Å)	15.6290(9)
c(Å)	13.1282(7)
α(°)	90.00
β(°)	118.2320(10)
γ(°)	90.00
V(Å^3)	2289.8(2)
Z	2
D_c(mg m^{-3})	1.287
θ range (°)	1.82–27.50
μ(mm^{-1})	0.172
Reflections collected	19652
Data/restraints/parameters	5242/0/298
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0614$, $\omega R_2 = 0.1938$
R indices (all data)	$R_1 = 0.0814$, $\omega R_2 = 0.2207$
CCDC	806204

137–138°C; FTIR ν 1672 (C═N), 1320 (C—N), 971 (CH═CH, trans) cm^{-1}; ^{1}H-NMR δ 7.03–7.64 (m, 20H, ArH), 6.85–6.91 (d, $J = 15.8$ Hz, 2H, benzothiazole ring—CH═), 6.51–6.55 (d, $J = 15.8$ Hz, 2H, benzene ring—CH═), 3.85 (s, 3H, —OCH_3), 2.10 (s, 6H, $COCH_3$); MS (ESI): *m/z* 697 ($M^+ + 1$); *Anal*. Calcd for $C_{42}H_{36}N_2O_4S_2$: C, 72.39; H, 5.21; N, 4.02; O, 9.18; S, 9.20; Found (%): C, 72.39; H, 5.20; N, 4.01; O, 9.18; S, 9.22.

General procedure for the synthesis of compounds 4a–c. To a stirred solution of compounds **2** (0.5 mmol) and benzohydroximinoyl chlorides (1.5 mmol) in CH_2Cl_2 (50 mL), a solution of Et_3N (1 mL) in the same solvent (10 mL) was added dropwise slowly. The mixture was kept under stirring for 36 h at RT. Then, the triethylamine hydrochloride byproduct was removed by filtration. Solvent was evaporated *in vacuo*, and the remaining materials were purified by silica gel column chromatography (ethylacetate/petroleum ether = 1:4, v/v) to afford the desired products **4a–c**.

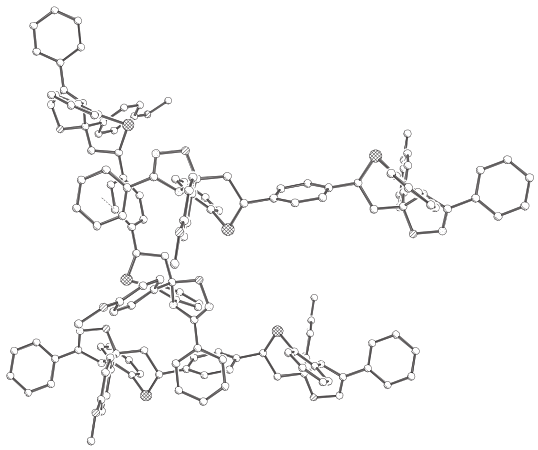


Figure 2. Weak offset face-to-face π···π interactions between cyclical 1 and cyclical 2.

2,2'-(1,4-Phenylene)-bis[3a-(4-phenyl)-4,5-dihydro-3aH-(3-phenyl-[1,2,4]oxadiazolo)-[5,4-d][1,5]benzothiazepine]. Pale yellow solid (23%); mp 302–304°C; FTIR ν 1605 (C═N), 1350 (C—N), 1249 (C—O—C), 693 (C—S—C) cm^{-1}; MS (EI): *m/z* 790 (M^+); *Anal*. Calcd for $C_{50}H_{38}N_4O_2S_2$: C, 75.92; H, 4.84; N, 7.08; O, 4.05; S, 8.11; Found (%):C, 75.93; H, 4.83; N, 7.08; O, 4.04; S, 8.12.

2,2'-(1,4-Phenylene)-bis[3a-(4-chlorophenyl)-4,5-dihydro-3aH-(3-phenyl-[1,2,4]oxadiazolo)-[5,4-d][1,5]benzothiazepine]. Pale white solid (20%); mp 322–324°C; FTIR ν 1606 (C═N), 1351 (C—N), 1249 (C—O—C), 692 (C—S—C) cm^{-1}; MS (EI): *m/z*

Table 2

Selected bond lengths (Å), bond angle (°), and torsion angle (°) of compound **4c**.

S(1)—C(25)	1.848(3)	C(1)—S(1)—C(25)	102.73(12)	N(2)—O(1)—C(17)—C(18)	83.2(2)
O(1)—C(17)	1.452(3)	C(26)—C(25)—C(30)	111.6(2)	C(17)—N(1)—C(8)—N(2)	158.6(2)
O(1)—N(2)	1.429(3)	C(8)—N(1)—C(17)	100.36(17)	C(25)—S(1)—C(1)—C(2)	−117.0(3)
N(1)—C(8)	1.429(3)	N(92)—O(1)—C(17)	104.9(2)	C(6)—N(1)—C(17)—C(30)	18.4(3)
N(1)—C(17)	1.474(3)	C(18)—C(17)—C(30)	113.00(19)	C(8)—N(1)—C(17)—C(18)	−84.6(2)

Table 3

Intermolecular interactions (Å) in compound **4c**.

D—H···A	D—H	H···A	D···A	∠D—H···A
C(3)—H(3)[a]···O(2)	0.930	2.522	3.392(4)	155.8
C(14)—H(14b)[b]···O(2)	0.930	2.649	3.374	135.3
C(28)—H(10B)[c]···Cg3	0.930	2.862	3.610	140.2

Cg3 is the center of gravity of ring C(9)—C(10)—C(11)—C(12)—C(13)—C(14)
[a] 1 − X, 1/2 + Y, 1/2 − Z
[b] 1 + X, Y, Z
[c] X, 1/2 − Y, 1/2 + Z

858 (M^+); *Anal*. Calcd for $C_{50}H_{36}Cl_2N_4O_2S_2$: C, 69.84; H, 4.22; Cl, 8.25; N, 6.52; O, 3.72; S, 7.46; Found (%): C, 69.84; H, 4.22; Cl, 8.25; N, 6.51; O, 3.73; S, 7.46.

2,2'-(1,4-Phenylene)-bis[3a-(4-methoxyphenyl)-4,5-dihydro-3aH-(3-phenyl-[1,2,4]oxadiazolo)-[5,4-d][1,5]benzothiazepine]. Colorless crystals (30%); mp 314–316°C; FTIR ν 1608 (C═N), 1351 (C—N), 1249 (C—O—C), 694 (C—S—C) cm^{-1}; MS (EI): *m/z* 850 (M^+); *Anal*. Calcd (%) for $C_{52}H_{42}N_4O_4S_2$: C, 73.39; H, 4.97; N, 6.58; O, 7.52; S, 7.54; Found (%): C, 73.39; H, 4.96; N, 6.58; O, 7.53; S, 7.54.

CONCLUSION

In summary, the present study described the synthesis of some novel 1,4-phenylene-bis[1,2,4]oxadiazolo[5,4-*d*][1,5] benzothiazepines (**4a–c**) via nitrile oxide cycloaddition. The bis[2-aryl-2′-(β-1,4-phenylenevinyl)-3-acetyl]-2,3-dihydro[1,5] benzothiazoles (**3a–c**) were also newly synthesized through ring contraction. The structural identities of compounds **4** was further confirmed by X-ray analysis of compound **4c** as typical example of the new bis-1,5-benzothiazepine derivatives with symmetrical structure.

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