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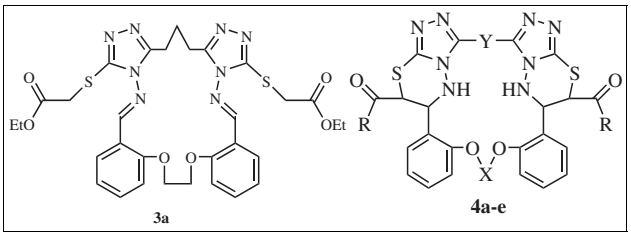

A practical and regioselective synthetic method for the synthesis of cis-diastereomers of bibracchial lariat ethers (BiBLEs) bearing ester and amide groups is reported. The novel BiBLEs **3a** and **4a–e** with neutral side arms were prepared by reaction of the corresponding aza-crown macrocycles **1a–c** with ethylchlroacetate and chloroacetamide. The structures of the new compounds have been confirmed by FTIR, ^{1}H, ^{13}C, DEPT, and MS spectroscopy.

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INTRODUCTION

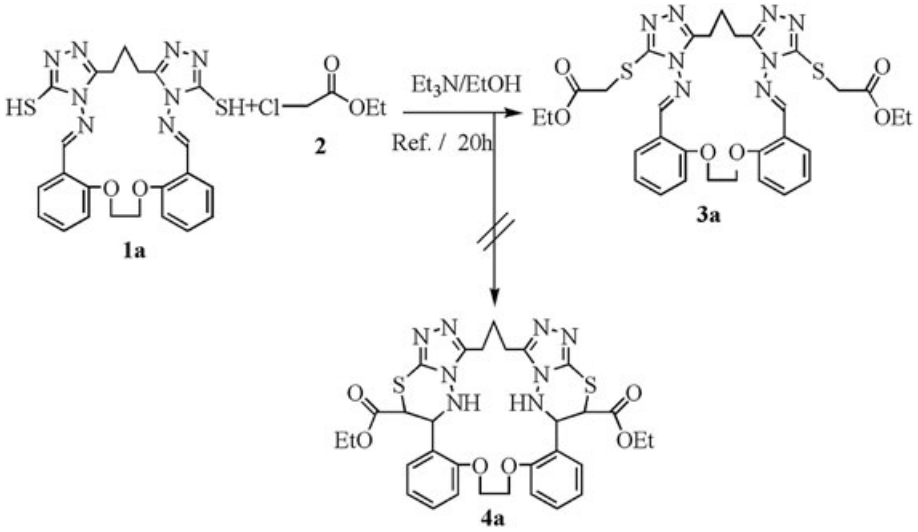
The incorporation of oxygen, nitrogen, and sulfur donor atoms in the structure of macrocycles will significantly affect their complexing properties because of the hard (O, N) and soft (S) character of the donor atoms and the exodentate tendency of the sulfide linkages [1,2]. Other changes involve the insertion of aromatic and/or heterocyclic ring systems into the macrocycles [3–5]; heterocyclic groups provide rigidity and are able in some cases to form complexes *via* their soft donor atoms [6]. The wide interest in the construction of synthetic macrocyclic compounds containing five- and six-membered heterocyclic rings as subunits has led to the preparation of a range of such compounds, which have been shown to possess very interesting properties in a variety of fields [7–10]. Lariat ethers are compounds with a macrocyclic ring and a side arm, which bears a donor group [11–13]. It has been designed as cation complexing agents, which exhibit complexation behavior similar to crown ethers, but with a three-dimensional binding character [14]. Lariat ethers can be divided into two categories depending on the nature of the secondary binding site that with a pendent neutral side arm and lariat ethers with a pendent proton-ionizable arm. Non-ionizable lariat ethers may exhibit an enhanced cation complexation and selectivity when compared with crown ethers without side arms [15].

The intensive development of the lariat crown ether concept has been directed toward the synthesis of several side armed crown ethers, designed for uses ranging from routine (polymer-supported PTC catalysts, separation/extraction reagents, etc.) to sophisticated applications as redox switches for membrane transport, synthetic cation conducting channels, nucleotide-based molecular boxes, and so on [16,17]. These valuable properties prompted us to synthesize some new series of 18–20 bibracchial lariat Ethers (BiBLEs) containing 1,3,4-thiadiazine-6-carboxylate or 1,3,4-thiadiazine-6-carboxamide.

RESULTS AND DISCUSSION

In continuation of our interst to develop the synthesis of new azathiacrown macrocycles and lariat ether [3,18], we report herein, a simple and efficient method for the regioselective synthesis of novel BiBLES **4a–e**. In this article, we demonstrate a novel method to introduce 1,2,4-triazolo[3,4-b][1,3,4]thiadiazines rings into macrocycles. The synthesis of BiBLEs containing 1,3,4-thiadiazine-6-carboxylate or 1,3,4-thiadiazine-6-carboxamide has not been reported yet. Aza-crown ether compounds **1a–c** were prepared according to the published method.[15] The functionalities in these aza-crown ethers made them valuable key precursors for the formation of different fused heterocyclic compounds. The available macrocycles **1a–c** encouraged us to study their transformation into the lariat ethers containing ester or amide groups. Thus, the novel lariat compounds **3a** and BiBLEs **4a–e** with neutral side arms were prepared by reaction of corresponding aza-crown macrocycles **1a–c** with ethylchlroacetate and chloroacetamide. The reactionof aza-crown macrocycle with ethylchloroacetate afforded different products depending on the base used.

Scheme 1. Synthesis of lariat ether **3a**.



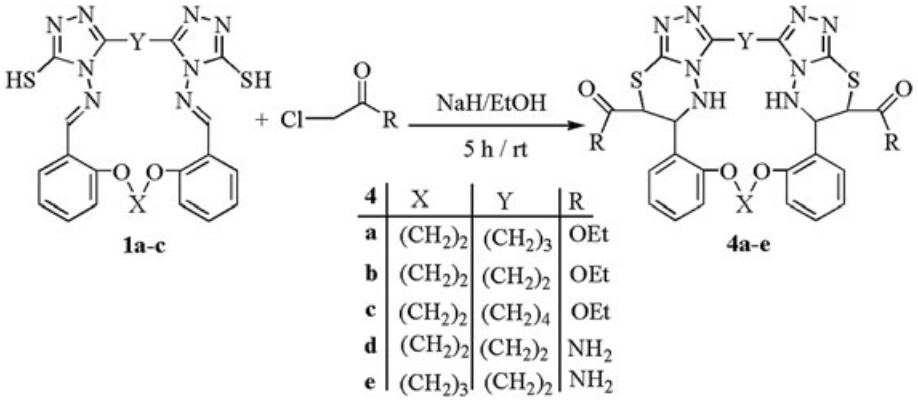
Initially, the reactivity of now available, aza-crown macrocycle with ethylchloroacetate in the present triethylamine as a base in refluxing ethanol was attempted. The reaction of compound **1a** with ethylchloroacetate under reflux conditions did not lead to the formation of BiBLE **4a**. Instead, the reaction gave another product, which could be characterized as the lariat ether **3a** (Schemes 1).

The formation of compound **3a** encouraged us to carry out this reaction in the presence of another base. Thus, stirring of compounds **1a–c** with ethylchloroacetat or chloroacetamide in the presence of sodium hydride for 5 h afforded 58–75% yields of the corresponding novel BiBLEs **4a–e** (Scheme 2).

The reaction proceeds *via* intramolecular cyclocondensation of the active methylene group with the imin group. The expected compounds were obtained in good yields. The reaction of **1a–c** with ethylchloroacetate and chloroacetamide in the presence sodium hydride was regioselective and afforded only cis isomer after ring closure. The isolated compounds **4a–e** were obtained as cis-diastereomers. This fact was confirmed by ^{1}H-NMR data. The stereochemistry of the products was determined from the coupling constant between two vicinal methine protons. In the ^{1}H-NMR spectra of compounds **4a–e**, the coupling constant ($^{3}J_{N—CH,\ CH—S} \approx 7.0–8.3$ Hz) is typical for the cis configuration [19–21].

The IR, ^{1}H–NMR, and ^{13}C-NMR spectra of **4a–e** confirmed the success of the cyclization by the disappearance of the signals corresponding to the SH and CH=N protons and the appearance of signals assigned to the methine and NH protons. The infrared spectra of the aza-crown **1a–c** showed absorptions band, at 2728 cm^{-1} due to SH groups, which were absent in the IR spectra of compounds **4a–e**. Similarly, the ^{1}H-NMR spectra of the compounds **1a–c** showed two characteristics absorption (singlet at: δ 8.3 ppm) attributed to the CH=N

Scheme 2. Synthesis of compounds **4a–e**.



4	X	Y	R
a	$(CH_2)_2$	$(CH_2)_3$	OEt
b	$(CH_2)_2$	$(CH_2)_2$	OEt
c	$(CH_2)_2$	$(CH_2)_4$	OEt
d	$(CH_2)_2$	$(CH_2)_2$	NH_2
e	$(CH_2)_3$	$(CH_2)_2$	NH_2

groups, and another at: δ 14.2 ppm, assigned to the SH, which were disappeared by the formation of compounds **4a**–**e**. In addition, the absence of the ^{13}C-NMR and DEPT signals due to the CH=N groups and appearance of the aliphatic carbon relative to the thiadiazine ring confirmed the formation of compounds **4a–e.**

CONCLUSIONS

In conclusion, we successfully prepared BiBLEs having pendant groups containing a strong donor group as a supporting ligand at the end of the sidearm.

EXPERIMENTAL

All products were characterized using IR, ^{1}H-NMR, ^{13}C-NMR spectra and the mass spectral data. All yields refer to isolated products. IR spectra were prepared on a galaxy series FTIR 5000 spectrophotometer using KBr discs. NMR spectra were recorded on Brucker spectrophotometer (300 MHZ) in DMSO-d_6 using TMS as an internal standard. Mass spectra were recorded on an Agilent Technology (HP) 5973 Network mass selective detector under electron impact (EI). C, H, N, and S analyses were performed on a Vario EL III elemental analyzer.

General procedure for the synthesis of compounds 3a. A mixture of compound **1a** (0.5 mmol) and triethylamine (3.0 mmol) in ethanol (5 mL) was maintained at reflux for 30 min. A solution of ethylchloroacetate (1.2 mmol) in ethanol (5 mL) was added, and the mixture was refluxed for 20 h. The reaction was monitored by TLC. After the completion of the reaction, the solvent was removed under reduced pressure. The crude solid was dissolved in $CHCl_3$, and the solution was washed with water. The organic layer was separated and dried over anhydrous sodium sulfate. The organic solvent was removed under reduced pressure. The residue was crystallized from ethylacetate to give the pure compound **3a**.

Ethyl-2-({9-[2ethoxy-2-oxoethyl)sulfanyl]-5,6,18,19-tetrahydro-4H-dibenzo[e,r]di[1,2,4]triazolo[4,3-i:3,4-n][1,4,8,9,15,16]dioxatetraazacyclononadecin-1-yl}sulfanyl)acetate 3a. 86% yield, IR (KBr): υ (cm^{-1}): 3062 (aromatic CH stretch.), 2937 (aliphatic CH stretch.) 1735 (C=O), 1601 (C=N), 1250, 1161, ^{1}H-NMR (300 MHz, DMSO d_6): δ 1.09 (t, 6H, $2CH_3$, J = 7.0 Hz), 2.05 (t, 2H, CH_2, J = 6.2 Hz), 2.77 (t, 4H, $2CH_2$, J = 6.1 Hz), 3.85 (s, 4H, $2SCH_2$), 3.98 (q, 4H, $2OCH_2$, J = 7.0 Hz), 4.52 (s, 4H, $2OCH_2$), 7.14 (t, 2H, $H_{arom.}$, J = 7.0 Hz), 7.27 (d, 2H, $H_{arom.}$, J = 8.3 Hz), 7.61 (d, 2H, $H_{arom.}$, J = 7.8 Hz), 7.75 (d, 2H, $H_{arom.}$, J = 7.6 Hz), 8.78 (s, 2H, 2CH=N), ^{13}C-NMR (75 MHz, DMSO-d_6): δ 14.3, 23.2, 24.9, 34.7, 61.6, 68.3, 114.5, 120.4, 121.9, 127.3, 135.5, 145.2, 152.6, 159.5, 162.0, 168.4. *Anal.* Calcd. for: $C_{31}H_{34}N_8O_6S_2$: C, 54.85; H, 5.05; N, 16.51; S, 9.45 Found: C, 54.57; H, 4.99; N, 16.31; S, 9.26.

General procedure for the synthesis of compounds 4a–e. Sodium hydride (2.5 mmol) was added to a solution of compounds **1a–c** (0.5 mmol) in absolute ethanol (10 mL) at room temperature. Salt formation was allowed to proceed at room temperature for 10 min and ethylchloroacetate or chloroacetamide (1.1 mmol) was added and the solution stirred for 5 h at room temperature. After the completion of the reaction, the solvent was removed under vacuum and extracted with ethylacetate; the organic layer was washed with water (3 × 10 mL), dried (Na_2SO_4), and evaporated under vacuum. The residue was crystallized from ethylacetate and petroleum ether to give compounds **4a–e.**

Diethyl-8,11-dioxa-32,36-dithia-19,20,22,23,29,30,34,35-octaazaheptacyclo[26.5.2.2^{18,21}.0^{2,7}.0^{12,17}.0^{20,24}.0^{31,35}]heptatriaconta-2,4,6,12(17),13,15,21,23,28,30-decaene-33,37-dicarboxylate (4a). *70% yield, IR (KBr): υ (cm^{-1}): 3250 (NH stretch.) 3064 (aromatic CH stretch.), 2955 (aliphatic CH stretch.) 1732 (C=O), 1610 (C=N), 1244, 1165, ^{1}H-NMR (300 MHz, DMSO d_6)*: δ 0.97 (t, 6H, $2CH_3$, J = 7.0 Hz), 2.00 (br, 2H, CH_2), 2.76 (br, 4H, $2CH_2$), 3.99 (q, 4H, $2CH_2$, J = 7.1 Hz), 4.35 (d, 2H, 2S—CH, J = 7.8 Hz), 4.44 (d, 2H, 2N—CH, J = 8.0 Hz), 4.78 (s, 4H, $2OCH_2$), 6.79 (d, 2H, $H_{arom.}$, J = 7.1 Hz), 7.02 (t, 2H, $H_{arom.}$, J = 7.0 Hz), 7.15 (d, 2H, $H_{arom.}$, J = 8.2 Hz), 7.30 (s, 2H, 2 NH·D_2O exchange), 7.34 (d, 2H, $H_{arom.}$, J = 5.0 Hz), ^{13}C-NMR (75 MHz, DMSO-d_6): δ 14.0, 23.1, 25.2, 40.3, 44.5, 61.8, 68.3, 113.4, 121.5, 124.1, 128.1, 130.8, 141.2, 152.5, 156.7, 168.9, DEPT: δ 14.0 (CH_3) 23.1 (CH_2), 25.2 (CH_2), 40.3 (CH), 44.5 (CH), 61.8 (CH_2), 68.3 (CH_2), 113.4 (CH), 121.5 (CH), 128.1 (CH), 130.8 (CH), m/z = 678 (M^+), 663, 648, 634, 588, 534, 324, 264, 146 (base peak), 120, 91. *Anal.* Calcd.for: $C_{31}H_{34}N_8O_6S_2$: C, 54.85; H, 5.05; N, 16.51; S, 9.45 Found: C, 54.51; H, 4.97; N, 16.33; S, 9.22.

Diethyl-8,11-dioxa-31,35-dithia-19,20,22,23,28,29,33,34-octaazaheptacyclo[25.5.2.2^{18,21}.0^{2,7}.0^{12,17}.0^{20,24}.0^{30,34}]hexatriaconta-2,4,6,12(17),13,15,21,23,27,29-decaene-32,36-dicarboxylate (4b). *70% yield, IR (KBr): υ (cm^{-1}): 3256 (NH stretch.) 3070 (aromatic CH stretch.), 2924 (aliphatic CH stretch.) 1730 (C=O), 1601 (C=N), 1248, 1161, ^{1}H-NMR (300 MHz, DMSO-d_6)*: δ 0.97 (t, 6H, $2CH_3$, J = 6.8 Hz), 2.93 (s, 4H, $2CH_2$), 3.97 (q, 4H, $2CH_2$, J = 6.5 Hz), 4.30 (d, 2H, 2S—CH, J = 8.1 Hz), 4.46 (d, 2H, 2N—CH, J = 8.3 Hz), 4.77 (s, 4H, $2OCH_2$), 6.72 (d, 2H, $H_{arom.}$, J = 7.2 Hz), 7.00 (t, 2H, $H_{arom.}$, J = 7.2 Hz), 7.13 (d, 2H, $H_{arom.}$, J = 8.0 Hz), 7.33 (s, 2H, 2 NH·D_2O exchange), 7.36 (d, 2H, $H_{arom.}$, J = 5.1 Hz), ^{13}C-NMR (75 MHz, DMSO-d_6): δ 14.0, 21.5, 40.1, 44.2, 62.1, 67.8, 113.0, 121.3, 124.6, 128.0, 130.6, 141.8, 152.9, 156.3, 168.8, DEPT: δ 14.0 (CH_3) 21.5 (CH_2), 40.2 (CH), 44.2 (CH), 62.2 (CH_2), 67.8 (CH_2), 113.0 (CH), 121.3 (CH), 128.0 (CH), 130.6 (CH), m/z = 664 (M+), 649, 634, 621, 606, 591, 577, 574, 551, 284, 264, 146 (base peak), 120, 91 *Anal.* Calcd. for: $C_{30}H_{32}N_8O_6S_2$: C, 54.20; H, 4.85; N, 16.86; S, 9.65. Found: C, 53.95; H, 4.75; N, 16.53; S, 9.46.

Diethyl-8,11-dioxa-33,37-dithia-19,20,22,23,30,31,35,36-octaazaheptacyclo[27.5.2.2^{18,21}.0^{2,7}.0^{12,17}.0^{20,24}.0^{32,36}]octatriaconta-2,4,6,12(17),13,15,21,23,28,31-decaene-34,38-dicarboxylate (4c). *58% yield, IR (KBr): υ (cm^{-1}): 3261 (NH stretch.) 3054 (aromatic CH stretch.), 2933, 2852 (aliphatic CH stretch.) 1732 (C=O), 1600 (C=N), 1240, 1161, ^{1}H-NMR (300 MHz, DMSO-d_6)*: δ 1.12 (br, 6H, $2CH_3$), 1.54 (br, 4H, $2CH_2$), 2.72 (s, 4H, $2CH_2$), 3.68 (q, 4H, $2CH_2$, J = 6.7 Hz), 4.38 (d, 2H, 2S—CH, J = 8.1 Hz), 4.49 (d, 2H, 2N—CH, J = 8.0 Hz), 4.65 (s, 4H, $2OCH_2$), 6.81 (d, 2H, $H_{arom.}$, J = 7.5 Hz), 7.04 (t, 2H, $H_{arom.}$, J = 7.0 Hz), 7.11 (d, 2H, $H_{arom.}$, J = 6.8 Hz), 7.25 (s, 2H, 2 NH·D_2O exchange), 7.30 (d, 2H, $H_{arom.}$, J = 5.7 Hz), ^{13}C-NMR (75 MHz, DMSO-d_6): δ 14.0, 23.0, 23.7 40.6, 44.0, 62.4, 68.8, 113.1, 121.0, 124.2, 128.1, 130.0, 141.5, 153.0, 156.1, 168.8, DEPT: δ 14.0 (CH_3) 23.0 (CH_2), 23.7 (CH_2), 40.6 (CH), 44.0 (CH), 62.4 (CH_2), 68.8 (CH_2), 113.1 (CH), 121.0 (CH), 128.1 (CH), 130.0; m/z = 692 (M+), 677, 634, 604, 284, 264, 146

(base peak), 120, 91. *Anal*. Calcd. for: $C_{32}H_{36}N_8O_6S_2$: C, 55.48; H, 5.24; N, 16.17; S, 9.26. Found: C, 55.13; H, 5.03; N, 15.98; S, 9.00.

8,11-Dioxa-31,35-dithia-19,20,22,23,28,29,33,34-octaazaheptacyclo[25.5.2.2^{18,21}.0^{2,7}.0^{12,17}.0^{20,24}.0^{30,34}]hexatriaconta-2,4,6,12(17),13,15,21,23,27,29-decaene-32,36-dicarboxamide (4d). *75% yield, IR (KBr): υ (cm^{-1}): 3315, 3184 (NH_2 stretch.) 3060 (aromatic CH stretch.), 2951 (aliphatic CH stretch.) 1689 (C=O), 1251, 1114, ^{1}H-NMR (300 MHz, DMSO-d_6)*: δ 2.90 (s, 4H, $2CH_2$), 4.22 (d, 2H, S—CH, J = 7.0 Hz), 4.43 (d, 2H, N—CH, J = 7.1 Hz), 4.61 (br, 2H, OCH_2), 4.77 (br, 2H, OCH_2), 6.62 (s, 2H, NH_2, D_2O exchange), 7.01 (t, 2H, $H_{arom.}$, J = 6.4 Hz), 7.12 (d, 2H, $H_{arom.}$, J = 7.5 Hz), 7.23 (s, 2H, 2 NH·D_2O exchange), 7.34 (t, 2H, $H_{arom.}$, J = 6.8 Hz), 7.43 (d, 2H, $H_{arom.}$, J = 5.7 Hz), 7.86 (s, 2H, NH_2, D_2O exchange), ^{13}C-NMR (75 MHz, DMSO-d_6): δ 21.7, 40.3, 43.7, 68.0, 113.1, 121.1, 124.8, 127.8, 130.5, 142.4, 152.8, 156.7, 169.6, DEPT: δ 21.7 (CH_2), 40.3 (CH), 43.7 (CH), 68.0 (CH_2), 113.0 (CH), 121.1 (CH), 127.8 (CH), 130.5 (CH), MS (EI): m/z = 606 (M^+), 577, 551, 395, 365, 339, 264, 146, 119, 91, 57, 43 (base peak). *Anal*. Calcd. for: $C_{26}H_{26}N_{10}O_6S_2$: C, 51.47; H, 4.32; N, 23.09; S, 10.57. Found: C, 51.23; H, 4.23; N, 22.86; S, 10.41.

8,12-Dioxa-32,36-dithia-20,21,23,24,29,30,34,35 octaazaheptacyclo[26.5.2.2^{19,22}.0^{2,7}.0^{13,18}.0^{21,25}.0^{31,35}]heptatriaconta-2,4,6,13(18),14,16,22,24,28,30-decaene-33,37-dicarboxamide (4e). *65% yield, IR (KBr): υ (cm^{-1}): 3330, 3176 (NH_2 stretch.) 3054 (aromatic CH stretch.), 2833–2941 (aliphatic CH stretch.) 1676 (C=O), 1242, ^{1}H-NMR (300 MHz, DMSO-d_6)*: δ 2.26 (br, 2H, CH_2), 3.00 (s, 4H, $2CH_2$), 4.19 (d, 2H, S—CH, J = 7.3 Hz), 4.49 (d, 2H, N—CH, J = 7.3 Hz), 4.62 (br, 2H, OCH_2), 4.79 (br, 2H, OCH_2), 6.70 (s, 2H, NH_2, D_2O exchange), 7.01 (t, 2H, $H_{arom.}$, J = 6.5 Hz), 7.14 (d, 2H, $H_{arom.}$, J = 8.1 Hz), 7.26 (s, 2H, 2 NH·D_2O exchange), 7.38 (t, 2H, $H_{arom.}$, J = 6.7 Hz), 7.56 (d, 2H, $H_{arom.}$, J = 5.9 Hz), 7.78 (s, 2H, NH_2, D_2O exchange), ^{13}C-NMR (75 MHz, DMSO-d_6): δ 21.3, 29.1, 40.1, 43.8, 65.3, 113.2, 120.9, 124.6, 128.0, 130.31, 142.46, 152.9, 156.7, 169.5, DEPT: δ 22.1 (CH_2), 22.8 (CH_2), 40.1 (CH), 43.8 (CH), 67.5 (CH_2), 112.9 (CH), 121.5 (CH), 128.0 (CH), 130.3 (CH), MS (EI): m/z = 620 (M^+), 590, 576, 532, 278, 146, 119, 57, 43 (base peak). *Anal*. Calcd. for: $C_{27}H_{28}N_{10}O_6S_2$: C, 52.25; H, 4.55; N, 22.57; S, 10.33. Found: C, 52.04; H, 4.39; N, 22.41; S, 10.18.

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