

A Convenient Synthesis of Thiamacrocylic Dilactams

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ABSTRACT: A convenient synthesis of 26- to 28-membered thiamacrocylic dilactams **8** was achieved via base-catalyzed condensation reaction of bis-2-cyanoacetamides **4** with dialdehyde derivatives **7**. The reaction was assumed to be geometrically stereoselective, affording *E*, *E'*-configuration as the only isolable isomer. *N*, *N'*-[Alkanediylbis(thia-2,1-phenylene)]-bis[2-cyanoacetamides] **4** were obtained by the reaction of cyanoacetic acid with the corresponding diamine hydrochlorides **3**. © 2007 Wiley Periodicals, Inc. Heteroatom Chem 18:249–254, 2007; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20292

INTRODUCTION

There is continuing interest in the preparation of macrocyclic diamides that have important uses in selective noble metal complexation [1–3] and metal ion selective electrodes [4,5] as well as being valuable intermediates for the synthesis of azacrowns and related compounds [6–10]. It has also been reported that the type of macrocyclic heteroatoms, number of amide groups, ring size, lipophilic groups as well as other structural features control their selectivity toward different ions [11–16]. For example, to improve the binding ability of macrocyclic receptors for alkali

metal cations, such attention has been paid to the development of functional groups in the ring [17–19] as incorporation of an amidic linkage in a polyether macrocycle may modify the binding properties of crown ether compounds to favor the alkali earth cations over alkali metal cations [20–24]. Gokel and coworkers have found that diaza-18-crown-6 derivatives with amide groups in their side chains exhibit extraordinary Ca²⁺ binding strength and remarkable selectivity for Ca²⁺ over Na⁺ [25], although a number of synthetic cyclopeptides are K⁺ or Ca²⁺ ionophores [26]. Moreover, some macrocyclic diamides have recently been used as new catalysts in the highly regioselective halogenation cleavage of epoxides with elemental halogens [27]. In addition, many reports of 1,4-dithia-12-crown-4 [28], hexathiacyclooctadecane [29] as well as *N,N'*-substituted 1,10-diaza-18-crown-6-ethers [30,31] were presented for use as Hg²⁺ electrodes.

Keeping the above facts in mind, it is intended in this investigation to report a convenient synthetic method for the preparation of benzo-fused thiamacrocylic dilactams. Utilization of easily accessible starting materials and facile synthetic approaches will also be taken into consideration.

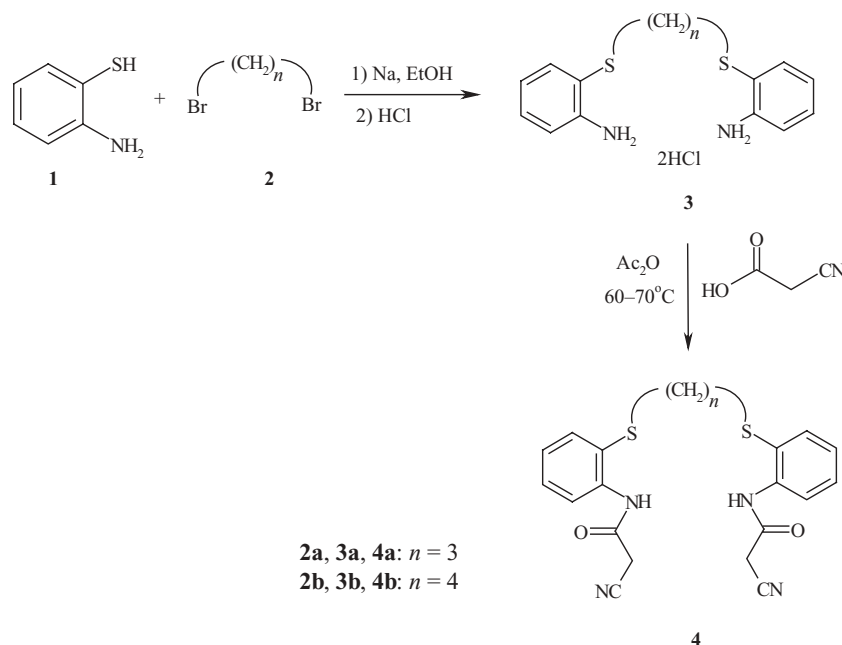
RESULTS AND DISCUSSION

2,2'-[Alkanediylbis(thio)]bisbenzenamines **3**, used essentially as the starting materials in this work, were prepared as hydrochloride salts from a modified simple synthetic method compared to the previously described ones [32,33]. Thus, the reaction

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SCHEME 1

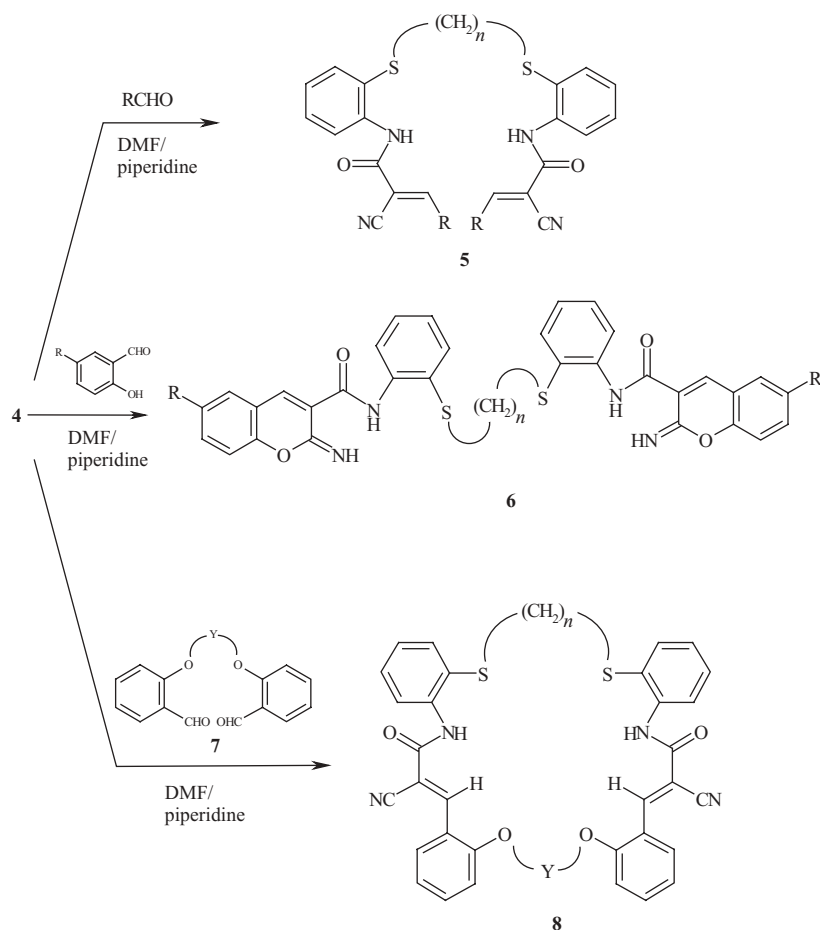
of 2-aminothiophenol (**1**) with dibromoalkanes **2a,b** in the presence of sodium ethoxide afforded the corresponding **3a,b** in good yields (71%–75%) after treatment with hydrochloric acid. Cyanoacetylation of many primary amine-containing compounds with cyanoacetic acid in acetic anhydride is a well-known synthetic tool for a long time [34]. Hence, the reaction of **3a,b** with cyanoacetic acid in warm acetic anhydride (60°C–70°C) gave *N,N'*-[alkanediylbis(thia-2,1-phenylene)]bis[2-cyanoacetamides] **4a,b**, whose structures were inferred through spectroscopic (IR, ^1H NMR, MS) and elemental analyses data. IR spectra of **4** exhibit, in addition to the carbonyl ($\nu = 1669\text{--}1657\text{ cm}^{-1}$) and nitrile ($\nu = 2257\text{--}2256\text{ cm}^{-1}$) stretching vibration bands, the amidic NH function at $\nu = 3273\text{--}3269\text{ cm}^{-1}$ region. ^1H NMR spectra of **4** reveal the activated methylene residue as a sharp singlet signal at $\delta = 3.60\text{--}3.97$ (Scheme 1) (Tables 1 and 2).

The corresponding ylidene derivatives obtained by the condensation reaction of cyanoacetamides with aromatic aldehydes under base-catalyzed conditions, involving the activated methylene function, have been studied intensively [35–38]. Thus, the reaction of the prepared 2-cyanoacetamide derivatives **4a,b** with aromatic aldehydes in *N,N'*-dimethylformamide in the presence of a catalytic amount of basic piperidine gave the corresponding arylidene derivatives **5a–e**. ^1H NMR spectra of **5** show the ylidene proton as a sharp singlet signal

at $\delta = 8.25\text{--}8.41$ region. The appearance of this signal at the mentioned chemical shift region confirms the presence of *2E,2'E*-configuration [36,39]. In other words, the reaction afforded only one isolable geometrically configurational isomer.

However, the reaction of **4a,b** with salicylaldehyde derivatives gave bis[2-imino-2*H*-1-benzopyran-3-carboxamides] **6a–d**, presumably via initial aldehydic condensation reaction with the active methylene function and subsequent hydroxyl nucleophilic attack at the nitrile residue. The disappearance of any band assignable to the nitrile group in IR spectra of **6** as well as the presence of two NH signals in ^1H NMR spectra at $\delta = 8.34\text{--}9.30$ and $12.56\text{--}12.87$ supports the assumed structure. In fact, formation of 3-substituted-2*H*-1-benzopyrans due to the base-catalyzed reaction of *o*-hydroxyaromatic aldehydes with cyanoacetic acid derivatives seems a general synthetic pathway toward the construction of this heterocyclic moiety [40].

On the other hand, the reaction of **4a,b** with bisaldehyde derivatives **7** in DMF in the presence of piperidine as the basic catalyst at room temperature successfully afforded the corresponding 26- to 28-membered diamide-containing macrocycles **8a–e**. ^1H NMR spectra of **8** reveal the ylidene singlet signals at $\delta = 8.55\text{--}8.86$. The downfield shift appearance of these protons compared with the case of **5** could be attributed to the effect of alkoxy group located at the ortho-position to the olefinic linkage [39] (Scheme 2) (Tables 1 and 2).



5a: R = Ph, $n = 3$

5b: R = Ph, $n = 4$

5c: R = 4-ClC₆H₄, $n = 3$

5d: R = 4-H₃COC₆H₄, $n = 3$

5e: R = 4-H₃COC₆H₄, $n = 4$

6a: R = H, $n = 3$

6b: R = Cl, $n = 3$

6c: R = H, $n = 4$

6d: R = Cl, $n = 4$

7a: Y = (CH₂)₃

7b: Y = (CH₂)₄

7c: Y =

8a: $n = 3$, Y = (CH₂)₃

8b: $n = 3$, Y = (CH₂)₄

8c: $n = 3$, Y =

8d: $n = 4$, Y = (CH₂)₃

8e: $n = 4$, Y = (CH₂)₄

SCHEME 2

Eventually, it could be concluded that an efficient synthetic approach was achieved for the desired macrocycles **8** from bis-2-cyanoacetamide-containing compounds **4** with dialdehydic derivatives **7** via condensation reaction of activated methylene residues. The reaction under the described conditions seems to be geometrically stereoselective, affording *E,E'*-configurational isomers.

EXPERIMENTAL

Melting points were recorded on an Electrothermal digital 9100 melting point apparatus, and are uncorrected. IR spectra were recorded (KBr) on a Perkin Elmer 1650 FT-IR spectrophotometer. ¹H NMR spectra were recorded on Varian GEMINI 200 (200 MHz) and Varian MERCURY 300 (300 MHz) spectrometers. Mass spectra were recorded on a Finnigan SSQ 7000 spectrometer (EI=70 eV).

TABLE 1 Physical Data of the Prepared Compounds

Compound	Melting Point (°C)(Solvent)	Yield (%) (Color)	Molecular Formula (Molecular Weight)	Analysis (%) Calculated/Found		
				C	H	N
3a	225–227	75 (colorless)	C ₁₅ H ₂₀ Cl ₂ N ₂ S ₂ (363.36)	49.58	5.55	7.71
				49.79	5.72	7.90
3b	253–255	71 (colorless)	C ₁₆ H ₂₂ Cl ₂ N ₂ S ₂ (377.39)	50.92	5.88	7.42
				51.01	5.99	7.34
4a	152–154 (acetic acid)	82 (colorless)	C ₂₁ H ₂₀ N ₄ O ₂ S ₂ (424.52)	59.41	4.75	13.20
				59.48	4.79	13.23
4b	159–161 (acetic acid)	78 (colorless)	C ₂₂ H ₂₂ N ₄ O ₂ S ₂ (438.55)	60.25	5.06	12.78
				60.27	5.08	12.72
5a	201–203 ^a	53 (almost colorless)	C ₃₅ H ₂₈ N ₄ O ₂ S ₂ (600.72)	69.97	4.70	9.33
				69.89	4.67	9.31
5b	226–228 ^a	52 (pale yellow)	C ₃₆ H ₃₀ N ₄ O ₂ S ₂ (614.75)	70.33	4.92	9.11
				70.31	4.91	9.14
5c	214–216 ^b	48 (pale yellow)	C ₃₅ H ₂₆ Cl ₂ N ₄ O ₂ S ₂ (669.62)	62.77	3.91	8.37
				62.72	3.88	8.32
5d	198–200 ^b	50 (pale yellow)	C ₃₇ H ₃₂ N ₄ O ₄ S ₂ (660.78)	67.25	4.88	8.48
				67.29	4.92	8.47
5e	210–212 ^a	48 (pale yellow)	C ₃₈ H ₃₄ N ₄ O ₄ S ₂ (674.80)	67.63	5.08	8.30
				67.65	5.11	8.32
6a	235–237 ^a	51 (almost colorless)	C ₃₅ H ₂₈ N ₄ O ₄ S ₂ (632.72)	66.44	4.46	8.86
				66.40	4.44	8.87
6b	194–196 ^b	45 (almost colorless)	C ₃₅ H ₂₆ Cl ₂ N ₄ O ₄ S ₂ (701.62)	59.91	3.74	7.99
				59.96	3.78	8.05
6c	226–228 ^a	49 (yellow)	C ₃₆ H ₃₀ N ₄ O ₄ S ₂ (646.75)	66.85	4.48	8.66
				66.86	4.69	8.62
6d	210–212 ^a	45 (pale yellow)	C ₃₆ H ₂₈ Cl ₂ N ₄ O ₄ S ₂ (715.64)	60.42	3.94	7.83
				60.38	3.91	7.82
8a	258–260 ^c	36 (yellow)	C ₃₈ H ₃₂ N ₄ O ₄ S ₂ (672.79)	67.83	4.79	8.33
				67.94	4.84	8.48
8b	256–258 ^b	35 (yellow)	C ₃₉ H ₃₄ N ₄ O ₄ S ₂ (686.81)	68.20	4.99	8.16
				68.40	5.09	8.05
8c	193–195 ^b	65 (yellow)	C ₄₃ H ₃₄ N ₄ O ₄ S ₂ (734.85)	70.28	4.66	7.62
				70.45	4.75	7.43
8d	233–235 ^a	47 (yellow)	C ₃₉ H ₃₄ N ₄ O ₄ S ₂ (686.81)	68.20	4.99	8.16
				68.11	4.87	8.09
8e	140–142 ^b	45 (yellow)	C ₄₀ H ₃₆ N ₄ O ₄ S ₂ (700.84)	68.55	5.18	7.99
				68.73	5.25	8.20

^a*N,N*-Dimethylformamids.^b*N,N*-Dimethylformamids/water as 9:1 v/v.^c*N,N*-Dimethylformamids/water as 4:1 v/v.

However, mass spectra of **8** were recorded on a Hewlett Packard model MS 5988 spectrometer (EI = 15 eV). The starting compounds **7a–c** [41] were prepared according to the previously reported procedures.

Synthesis of 2,2'-[Alkanediylbis(thio)]-bisbenzenamine dihydrochlorides **3a,b**

A solution of dibromoalkane **2a,b** (50 mmol) in absolute ethanol (10 mL) was added dropwise within a 15-min period to a refluxing solution of 2-aminothiophenol **1** (100 mmol) in absolute ethanol (100 mL) containing sodium (100 mmol). After com-

plete addition, the solution was refluxed for 1 h, and then the reaction mixture was poured onto ice-cold water (500 mL). The separated oily mass was dissolved in methanol (100 mL), and then acidified with concentrated HCl (33%). The separated colorless solid was collected, washed with diethyl ether, and used as it is described in the following steps without any further purification.

Synthesis of *N,N'*-[Alkanediylbis(thia-2,1-phenylene)]bis[2-cyanoacetamides] **4a,b**

A mixture of the appropriate **3a,b** (10 mmol) and cyanoacetic acid (20 mmol) in acetic anhydride

TABLE 2 Spectroscopic Data of the Prepared Compounds

Compound	IR (ν_{max}/cm^{-1})	1H NMR (Solvent), δ	MS, m/z (%)
3a	3423, 3109, 1551, 1470, 1409	(D ₂ O) 1.85 (quintet, $J = 7.5$ Hz, 2H, SCH ₂ CH ₂), 3.07 (t, $J = 7.5$ Hz, 4H, 2SCH ₂), 7.41–7.61 (m, 8H, arom. H)	290 [(M – 2HCl), 100]
3b	3446, 3147, 3093, 1552, 1473, 1413	(D ₂ O) 1.70 (br. s, 4H, 2SCH ₂ CH ₂), 2.98 (br. s, 4H, 2SCH ₂), 7.47–7.68 (m, 8H, arom. H)	304 [(M – 2HCl), 23]
4a	3273, 2257, 1669, 1578, 1534	(CDCl ₃) 1.81 (quintet, $J = 7$ Hz, 2H, SCH ₂ CH ₂), 2.88 (t, $J = 7$ Hz, 4H, 2SCH ₂), 3.60 (s, 4H, 2COCH ₂), 7.06–8.31 (m, 8H, arom. H), 9.13 (br. s, 2H, 2NH)	424 (M, 100)
4b	3269, 2256, 1657, 1585, 1532	(DMSO- <i>d</i> ₆) 1.68 (br. s, 4H, 2SCH ₂ CH ₂), 2.93 (br. s, 4H, 2SCH ₂), 3.97 (s, 4H, 2COCH ₂), 7.22–7.50 (m, 8H, arom. H), 9.76 (s, 2H, 2NH)	438 (M, 19)
5a	3305, 2204, 1683, 1578, 1520	(CDCl ₃) 1.80 (quintet, $J = 6.8$ Hz, 2H, SCH ₂ CH ₂), 2.86 (t, $J = 6.8$ Hz, 4H, 2SCH ₂), 7.00–7.94 (m, 16H, arom. H), 8.32 (s, 2H, 2 olefinic H), 8.42 (d, $J = 7.2$ Hz, 2H, arom. H), 9.59 (s, 2H, 2NH)	600 (M, 18)
5b	3302, 2205, 1686, 1581, 1522	(CDCl ₃) 1.72 (br. s, 4H, 2SCH ₂ CH ₂), 2.79 (br. s, 4H, 2SCH ₂), 7.06–8.02 (m, 16H, arom. H), 8.41 (s, 2H, 2 olefinic H), 8.50 (dd, $J = 1.2, 8.2$ Hz, 2H, arom. H), 9.68 (s, 2H, 2NH)	614 (M, 2)
5c	3290, 2211, 1692, 1583, 1525	(CDCl ₃) 1.79 (quintet, $J = 7.2$ Hz, 2H, SCH ₂ CH ₂), 2.85 (t, $J = 7.2$ Hz, 4H, 2SCH ₂), 7.00–7.87 (m, 14H, arom. H), 8.25 (s, 2H, 2 olefinic H), 8.40 (dd, $J = 1.0, 8.8$ Hz, 2H, arom. H), 9.58 (s, 2H, 2NH)	668 (M, 17)
5d	3285, 2203, 1679, 1582, 1517	(CDCl ₃) 1.87 (quintet, $J = 7.2$ Hz, 2H, SCH ₂ CH ₂), 2.93 (t, $J = 7.4$ Hz, 4H, 2SCH ₂), 3.91 (s, 6H, 2OCH ₃), 6.99–8.02 (m, 14H, arom. H), 8.31 (s, 2H, 2 olefinic H), 8.49 (dd, $J = 1.2, 8.2$ Hz, 2H, arom. H), 9.61 (s, 2H, 2NH)	660 (M, 5)
5e	3291, 2203, 1680, 1587, 1520	(CDCl ₃) 1.73 (br. s, 4H, 2SCH ₂ CH ₂), 2.79 (br. s, 4H, 2SCH ₂), 3.90 (s, 6H, 2OCH ₃), 6.99–8.03 (m, 14H, arom. H), 8.33 (s, 2H, 2 olefinic H), 8.50 (dd, $J = 1.5, 8.4$ Hz, 2H, arom. H), 9.62 (s, 2H, 2NH)	674 (M, 2)
6a	3316, 1678, 1577, 1532	(DMSO- <i>d</i> ₆) 1.70 (quintet, $J = 7$ Hz, 2H, SCH ₂ CH ₂), 2.97 (t, $J = 7$ Hz, 4H, 2SCH ₂), 6.86–8.55 (m, 18H, arom. H), 9.14 (s, 2H, 2NH), 12.87 (s, 2H, 2NH)	
6b	3259, 1681, 1579, 1537	(CDCl ₃) 1.72 (quintet, $J = 7$ Hz, 2H, SCH ₂ CH ₂), 2.85 (t, $J = 7$ Hz, 4H, 2SCH ₂), 6.93–8.43 (m, 18H, 16 arom. H + 2NH), 12.56 (s, 2H, 2NH)	
6c	3293, 1677, 1604, 1574	(DMSO- <i>d</i> ₆) 1.63 (br. s, 4H, 2SCH ₂ CH ₂), 2.84 (br. s, 4H, 2SCH ₂), 7.05–8.56 (m, 18H, arom. H), 9.18 (s, 2H, 2NH), 12.84 (s, 2H, 2NH)	
6d	3316, 1678, 1580, 1534	(DMSO- <i>d</i> ₆) 1.62 (br. s, 4H, 2SCH ₂ CH ₂), 2.83 (br. s, 4H, 2SCH ₂), 7.05–8.53 (m, 16H, arom. H), 9.30 (s, 2H, 2NH), 12.78 (s, 2H, 2NH)	
8a	3316, 3288, 2205, 1686, 1582, 1517	(CDCl ₃) 1.88 (quintet, $J = 7$ Hz, 2H, SCH ₂ CH ₂), 2.40 (quintet, $J = 5.2$ Hz, 2H, OCH ₂ CH ₂), 2.83 (t, $J = 7.4$ Hz, 4H, 2SCH ₂), 4.42 (t, $J = 5.4$ Hz, 4H, 2OCH ₂), 7.05–8.34 (m, 16H, arom. H), 8.81 (s, 2H, 2 olefinic H), 9.61 (s, 2H, 2NH)	672 (M, 61)
8b	3304, 2204, 1687, 1583, 1523	(The product is insoluble in deuterated solvents)	687 [(M + 1), 28]
8c	3291, 2205, 1687, 1581, 1521	(DMSO- <i>d</i> ₆) 1.64 (quintet, $J = 6.6$ Hz, 2H, SCH ₂ CH ₂), 2.86 (t, $J = 6.6$ Hz, 4H, 2SCH ₂), 5.33 (s, 4H, 2 ArCH ₂ O), 7.02–8.03 (m, 20H, arom. H), 8.57 (s, 2H, 2 olefinic H), 9.67 (s, 2H, 2NH)	735 [(M + 1), 12]
8d	3281, 2204, 1683, 1579, 1522	(DMSO- <i>d</i> ₆) 1.59 (br. s, 4H, 2SCH ₂ CH ₂), 2.30 (br. s, 2H, OCH ₂ CH ₂), 2.79 (br. s, 4H, 2SCH ₂), 4.30 (t, $J = 4.8$ Hz, 4H, 2OCH ₂), 7.02–8.10 (m, 16H, arom. H), 8.55 (s, 2H, 2 olefinic H), 9.75 (s, 2H, 2NH)	687 [(M + 1), 27]
8e	3292, 2206, 1686, 1579, 1520	(CDCl ₃) 1.70 (br. s, 4H, 2SCH ₂ CH ₂), 2.11 (br. s, 4H, 2OCH ₂ CH ₂), 2.77 (br. s, 4H, 2SCH ₂), 4.18 (br. s, 4H, 2OCH ₂), 6.95–8.51 (m, 16H, arom. H), 8.86 (s, 2H, 2 olefinic H), 9.60 (s, 2H, 2NH)	700 (M, 27)

(20 mL) was stirred at 60°C to 70°C for 3 h. The reaction mixture was poured onto cold water (200 mL). The separated solid was collected, washed with water, and finally crystallized from acetic acid affording colorless crystals of **4a,b**.

Reaction of **4a,b** With Aromatic Aldehyde

A mixture of the appropriate **4a,b** (1.25 mmol) and the corresponding aldehyde (2.5 mmol) in *N,N'*-dimethylformamide (10 mL) containing piperidine (2–3 drops) was stirred at room temperature

(25°C–30°C) for 24 h. The solid separated was collected and crystallized from a suitable solvent affording **5a–e** and **6a–d**.

Synthesis of Thiamacrocylic Diamides **8a–e**

A mixture of equimolar amounts of **4a,b** and the corresponding **7a–c** (1.25 mmol) in *N,N*-dimethylformamids (10 mL) containing piperidine (2–3 drops) was stirred at room temperature (20°C–25°C) for 2, 4, and 7 days in case of **8c**, **8d**, and **8a,b,e**, respectively. The separated solid was collected and crystallized from a suitable solvent affording **8a–e**.

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