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Regiocontrolled Syntheses of Some New Spiro[polycyclic-1′-isothiochroman]-4′-one Derivatives

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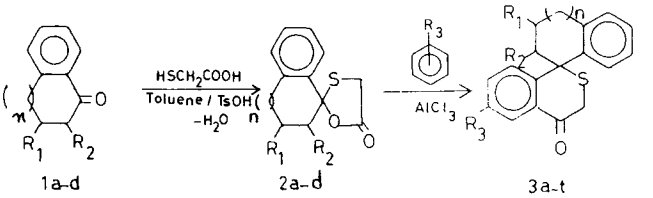
ABSTRACT

*Indan-1-one (**1a**), 1-tetralone (**1b**), fluorenone (**1c**), and anthrone (**1d**) reacted with mercaptoacetic acid in toluene in the presence of p-toluenesulfonic acid to give spiro[indan-1,2′-[1′,3′]oxathialan]-5′-one (**2a**), spiro[tetrahydro-naphthalene-1,2′-[1,3′]oxathialan]-5′-one (**2b**), spiro[fluorene9,2′-[1′,3′]-oxathialan]-5′-one (**2c**), and spiro[anthracene-9(10H)-2′-[1′,3′]-oxathialan]-5′-one (**2d**), respectively. Compounds **2a–d** reacted with arenes in the presence of aluminum chloride to yield spiro[polycyclic-1′-isothiochroman]-4′-one derivatives **3a–t**. The mechanisms of these reactions are discussed. All the synthesized spiroheterocycle derivatives were identified by conventional methods (IR, ^{1}H-NMR spectroscopy) and elemental analyses. © 1996 John Wiley & Sons, Inc.*

INTRODUCTION

Several authors have recently reported the syntheses and applications of spirocyclic derivatives [1–8]. Also, a facile access to aphidicolane and stemodane B/C/D-ring systems was reported [9], and the syntheses of some new heterobicyclic compounds containing the spiro-1,2,4-triazine moiety as potential anti-HIV and anticancer agents were investigated [10].



1 & 2	R_1	R_2	n
a	H	H	0
b	H	H	1
c			0
d			1

3a-t	R_1	R_2	R_3	n
a	H	H	H	0
b	H	H	CH_3	0
c	H	H	OCH_3	0
d	H	H	Br	0
e	H	H		0
f	H	H	H	1
g	H	H	CH_3	1
h	H	H	OCH_3	1
i	H	H	Br	1
j	H	H		1
k			H	0
l			CH_3	0
m			OCH_3	0
n			Br	0
o				0
p			H	1
q			CH_3	1
r			OCH_3	1
s			Br	1
t				1

SCHEME 1

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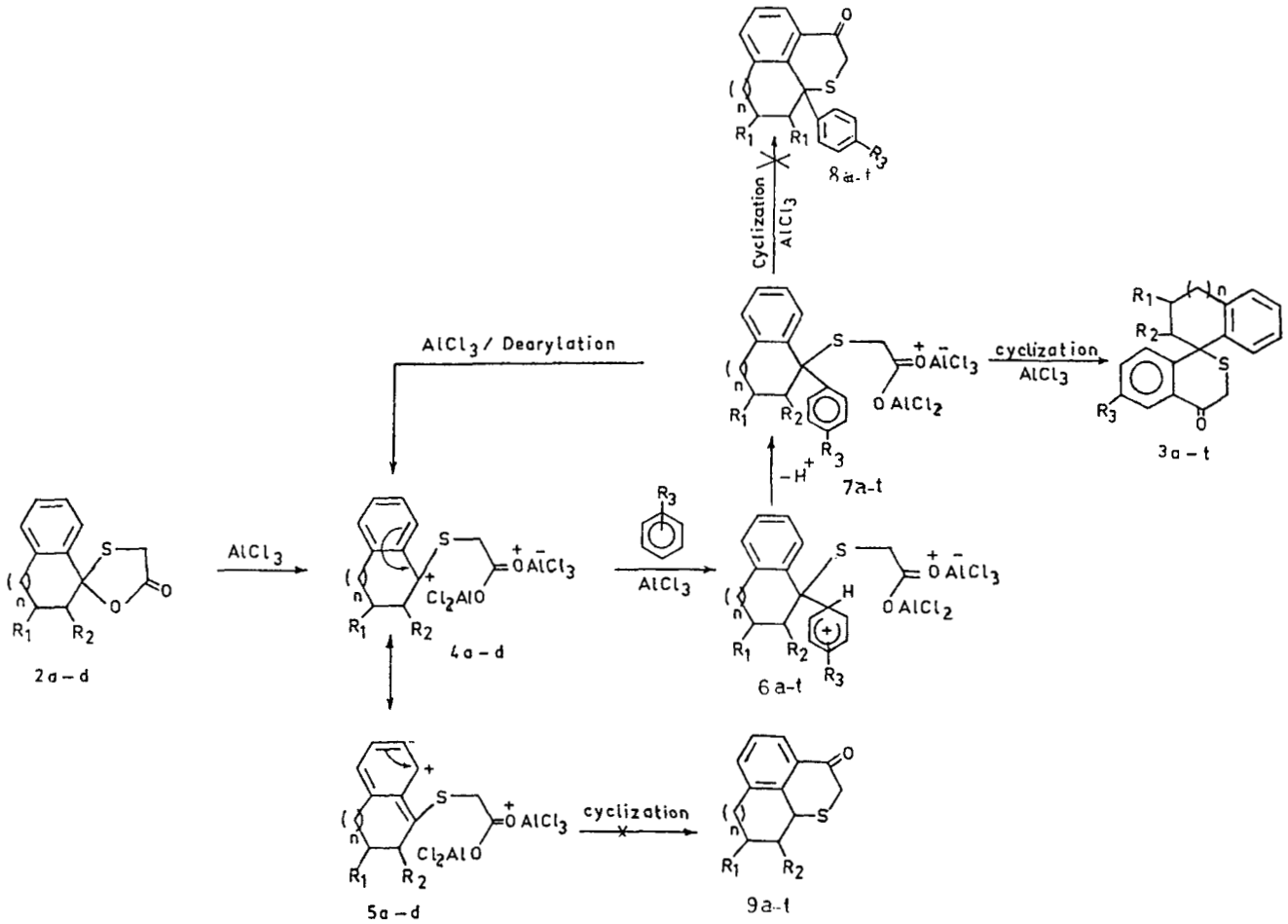

SCHEME 2

Spiroheterocycles have also been prepared to be used as herbicides and pesticides [11], as aldose reductase inhibitors [12] and for biological testing [13]. The preparation of fluoran compounds for use as recording materials has been carried out [14–16]. The syntheses of spiroheterocycles using different techniques and catalysts have been accomplished [17–27]. Conformational analysis of spiroisothiochromanes using NMR spectroscopy and X-ray spectroscopy has been investigated [28]. From all the foregoing facts, and as a continuation of our previous work [29,30], together with our interest in Friedel-Crafts chemistry [31–35] and the synthesis of spiroheterocycles [36–40], we report herein the syntheses of some new spiro[polycyclic-1′-isothiochroman]-4′-one derivatives.

RESULTS AND DISCUSSION

The syntheses of the useful spiroheterocycles [1–16] have gained some importance. In some cases, these syntheses required several steps [1–16]. Our syntheses were targeted for the synthesis of new spiro[polycyclic-1′-isothiochroman]-4′-one derivatives **3a–t** that are analogous to some well-known biologically active spiro derivatives [1–8]. The advantages of our methods were the use of inexpensive precursors and facile reactions with readily available reagents and simple techniques.

Our syntheses were initiated with the reaction of 1-indanone (**1a**), 1-tetralone (**1b**), fluorenone (**1c**), and anthrone (**1d**) with mercaptoacetic acid in the presence of *p*-toluenesulfonic acid in toluene to afford spiro[indan-1,2′-[1′,3′]oxathialan]-5′-one (**2a**, 86%), spiro[tetrahydronaphthalene-1,2′-[1′,3′]-oxathialan]-5′-one (**2b**, 88%), spiro[fluorene-9,2′-[1′,3′]oxathialan]-5′-one (**2c**, 95%) and spiro[anthracene-9(10*H*), 2′-[1′,3′]oxathialan]-5′-one (**2d**, 98%) (Scheme 1). Reactions of compounds **2a–d** with arenes in the presence of aluminum chloride gave spiro[polycyclic-1′-isothiochroman]-4′-one derivatives **3a–t** in fairly good yields (55–70%) (Scheme 1). For instance, the reaction of benzene with **2a** gave spiro[indan-1,1′-isothiochroman]-4′-one **3a** in 55%

TABLE 1 Physical Data of Spiro[polycyclic-2′[1′,3′]oxathialan]-5′-ones **2a–d** and Spiro[polycyclic-1′-isothiochroman]-4′-one Derivatives **3a–t**

Compound No.	*Yield (%)*	*MP (°C)*	*Molecular Formula (Solvent of Crystallization)*	*IR (K, Br), cm^{-1}*	*^1H NMR (Solvent), δ(TMS)*
2a	86	120–122	$C_{11}H_{10}O_2S$(toluene)	3030(CH arom), 2850-(CH aliph), 1725(C=O), 720(C–S)	(DMSO-d_6) 2.00–2.40(4H, m), 3.38(2H, s), 7.00–7.50(4H, m)
2b	88	160–162	$C_{12}H_{12}O_2S$(toluene)	3050(CH arom), 2860-(CH aliph), 1720(C=O), 730(C–S)	(DMSO-d_6) 2.00–2.50(6H, m), 3.40(2H, s), 7.00–7.50(4H, m)
2c	95	192–194	$C_{15}H_{10}O_2S$(toluene)	3080(CH arom), 2870-(CH aliph), 1750(C=O), 710(C–S)	(DMSO-d_6) 3.50(2H, s), 7.00–7.60(8H, m)
2d	98	220–222	$C_{16}H_{12}O_2S$(toluene)	3050(CH arom), 2860-(CH aliph), 1720(C=O), 730(C–S)	(DMSO-d_6) 3.40(2H, s), 3.30(2H, s), 7.00–7.80-(8H, m)
3a	55	90–92	$C_{17}H_{14}OS$(ethanol)	3080(CH arom), 2850-(CH aliph), 1715(C=O), 710(C–S)	(DMSO-d_6) 2.00–2.40(4H, m), 3.39(2H, s), 7.00–7.60(8H, m)
3b	63	94–96	$C_{18}H_{16}OS$(ethanol)	3030(CH arom), 2880-(CH aliph), 1710(C=O), 730(C–S)	(DMSO-d_6) 2.00–2.40(4H, m), 2.30(3H, s), 3.40(2H, s), 7.00–7.60-(7H, m)
3c	62	118–120	$C_{18}H_{16}O_2S$(ethanol)	3030(CH arom), 2880-(CH aliph), 1715(C=O), 720(C–S)	(DMSO-d_6) 2.00–2.20(4H, m), 3.40(3H, s), 7.00–7.40(7H, m)
3d	57	120–122	$C_{17}H_{13}OSBr$(acetic acid/water 1:1)	3020(CH arom), 2890-(CH aliph), 1710(C=O), 730(C–S)	(DMSO-d_6) 2.00–2.40(4H, m), 3.45(2H, s), 7.00–7.40(7H, m)
3e	66	100–102	$C_{23}H_{18}OSr$(ethanol)	3080(CH arom), 2880-(CH aliph), 1715(C=O), 720(C–S)	(DMSO-d_6) 2.00–2.40(4H, m), 3.35(2H, s), 7.00–8.00(12H, m)
3f	65	115–117	$C_{18}H_{16}OS$(ethanol/water 1:1)	3080(CH arom), 2900-(CH aliph), 1705(C=O), 710(C–S)	($CDCl_3$) 1.80–2.10(2H, m), 2.40–2.60(2H, t), 2.70–2.85(2H, t), 3.40-(2H, s), 7.00–7.80(8H, m)
3g	62	120–122	$C_{19}H_{18}OS$(ethanol/water 4:1)	3070(CH arom), 2858-(CH aliph), 1700(C=O), 730(C–S)	($CDCl_3$) 1.80–2.10(2H, m), 2.30(3H, s), 2.40–2.60(2H, t) 2.70–2.80(2H, t), 3.38-(2H, s), 7.00–7.70(7H, m)
3h	62	130–132	$C_{19}H_{18}O_2S$(methanol/water 4:1)	3070(CH arom), 2860-(CH aliph), 1700(C=O), 720(C–S)	($CDCl_3$) 1.80–2.00(2H, m), 2.40–2.60(2H, t), 2.75–2.85(2H, t), 3.20(3H, s), 3.40(2H, s), 7.00–7.80(7H, m)
3i	62	110–112	$C_{18}H_{15}OSBr$(dilute acetic acid)	3060(CH arom), 2860-(CH aliph), 1710(C=O), 730(C–S)	($CDCl_3$) 1.80–2.10(2H, m), 2.40–2.60(2H, t), 2.70–2.90(2H, t), 3.35(2H, s), 7.00–7.80-(7H, m)
3j	66	122–124	$C_{24}H_{20}OS$(ethanol)	3060(CH arom), 2870-(CH aliph), 1710(C=O), 730(C–S)	($CDCl_3$) 1.80–2.10(2H, m), 2.40–2.60(2H, t), 2.70–2.80(2H, t), 3.40(2H, s), 7.00–8.20-(12H, m)
3k	62	160–162	$C_{21}H_{14}OS$(acetic acid/water 3:1)	3080(CH arom), 2860-(CH aliph), 1710(C=O), 730(C–S)	(DMSO-d_6) 3.35(2H, s), 7.00–8.10-(12H, m)
3l	70	164–166	$C_{22}H_{16}OS$(ethanol)	3050(CH arom), 2900-(CH aliph), 1705(C=O), 720(C–S)	(DMSO-d_6) 2.30(3H, s), 3.70(2H, s), 7.00–7.85-(11H, m)

TABLE 1 continued Physical Data of Spiro[polycyclic-2′[1′,3′]oxathialan]-5′-ones **2a–d** and Spiro[polycyclic-1′-isothiochroman]-4′-one Derivatives **3a–t**

Compound No.	*Yield (%)*	*MP (°C)*	*Molecular Formula (Solvent of Crystallization)*	*IR (K, Br), cm⁻¹*	*¹H NMR (Solvent), δ(TMS)*
3m	68	170–172	$C_{22}H_{16}O_2S$(ethanol)	3020(CH arom), 2890-(CH aliph), 1710(C=O), 720(C–S)	(DMSO-d_6) 3.20(3H, s), 3.70(2H, s), 7.00–7.90-(11H, m)
3n	55	180–182	$C_{21}H_{13}OSBr$(acetic acid/water 4:1)	3050(CH arom), 2880-(CH aliph), 1710(C=O), 730(C–S)	(DMSO-d_6) 3.70(2H, s), 7.00–7.70(11H, m)
3o	61	188–190	$C_{27}H_{18}OS$(ethanol)	3050(CH arom), 2890-(CH aliph), 1710(C=O), 710(C–S)	(DMSO-d_6) 3.50(2H, s), 7.00–8.10(16H, complex)
3p	64	200–202	$C_{22}H_{16}OS$(acetic acid/water 1:1)	3050(CH arom), 2900-(CH aliph), 1710(C=O), 710(C–S)	(DMSO-d_6) 3.50(2H, s), 4.10(2H, s), 7.00–8.10-(12H, m)
3q	66	170–172	$C_{23}H_{18}OS$(ethanol)	3020(CH arom), 2900-(CH aliph), 1710(C=O), 720(C–S)	(DMSO-d_6) 2.30(3H, s), 3.50(2H, s), 4.10(2H, s), 7.00–8.00(11H, m)
3r	69	180–182	$C_{23}H_{18}O_2S$(ethanol)	3050(CH arom), 2890-(CH aliph), 1710(C=O), 710(C–S)	(MDSO-d_6) 3.20(3H, s), 3.50(2H, s), 4.20(2H, s), 7.00–8.10(11H, m)
3s	56	240–242	$C_{22}H_{15}OSBr$(acetic acid/water 2:1)	3050(CH arom), 2890-(CH aliph), 1710(C=O), 720(C–S)	(DMSO-d_6) 3.50(3H, s), 4.20(2H, s), 7.00–7.80(11H, m)
3t	57	260–262	$C_{28}H_{18}OS$(ethanol)	3030(CH arom), 2890-(CH aliph), 1710(C=O), 710(C–S)	(MDSO-d_6) 3.50(2H, s), 4.30(2H, s), 7.00–7.90-(16H, m)

[a]All the prepared compounds gave satisfactory elemental analyses.

yield. When **2b** reacted with toluene, 6-methylspiro[tetrahydronaphthalene-1,1′-isothiochroman]-4′ -one (**3g**) was formed in 62% yield. Also, the reaction of **2c** and **2d** with anisole yielded 6-methoxyspiro[flourene-9,1′-isothiochroman]-4′-one **3m** and 6-methoxyspiro[anthracene-9 (10H)-1′-isothiochroman]-4′-one **3r** in 68% and 69% yields, respectively. The formation of compounds **3a–t** could be explained as shown in Scheme 2. It is clear from Scheme 2 that spiro[polycyclic-2-[1′,3′]oxathialan]-5′-one derivative **2a–d** reacted with $AlCl_3$ to form the carbocations **4a–d**. The carbocations **4a–d** reacted with arenes in the presence of $AlCl_3$ to yield the intermediates **6a–t**, which underwent deprotonation to afford **7a–t**. Compounds **7a–t** are cyclized in the presence of aluminum chloride to give the new target compounds spiro[polycyclic-1′-isothiochroman]-4′-ones **3a–t** (Scheme 2).

The failure to detect the expected cyclization products **4a–t** was based on the fact that the intermediates **5a–d** with a positive charge in the aromatic ring never cyclize because the Friedel-Crafts reactions are electrophilic reactions (Scheme 2). The preferential cyclization of compounds **7a–t** to give **3a–t** and the failure to detect the expected cyclization products **8a–t** were attributed to the relative stabilities of **3a–t** compared with **8a–t** under the Friedel-Crafts reaction conditions [41]. The structures of compounds **2a–d** were established from their elemental analyses and spectroscopic data (Table 1).

The IR spectrum of compound **2a** showed the following absorption bands: 3050 cm^{-1} for aromatic CH, 2880 cm^{-1} for aliphatic CH, 1725 cm^{-1} for the carbonyl group, and 730 cm^{-1} for C–S stretching. The ^{1}H-NMR spectrum of **2a** (DMSO-d_6/TMS) showed the following signals: δ 2.00–2.40 (4H, m) for the two methylene groups of the indan ring, 4.00 (2H, s) for the methylene protons of the oxathialan ring, and 7.00–7.50 (4H, m) for the aromatic protons of the indan ring. Also, the structures of compounds **3a–t** were elaborated from their elemental analyses and spectroscopic data (Table 1). The IR spectrum of **3a** showed the following absorption bands: 3080 cm^{-1} for aromatic CH, 2880 cm^{-1} for the aliphatic CH stretching vibration, and 1715 cm^{-1} for the carbonyl group stretching vibrations. Also the ^{1}H-NMR spectrum of **3a** (DSMO-d_6/TMS) showed the following signals: δ 2.00–2.40 (4H, m) for the protons of the two methylene groups of the indan ring, 3.39 (2H, s) for the methylene protons of the isothochroman ring at C_3, and 7.00–7.60 (8H, m) for the aromatic protons of the indan and isothiochroman rings.

EXPERIMENTAL

The time required for completion of the reaction was monitored by thin-layer chromatography (TLC).

Melting points are uncorrected. ^{1}H-NMR spectra were measured on a Varian EM-360 90-MHz spectrometer. Infrared spectra were recorded on a Pye-Unicam SP 200-G spectrometer. Elemental analyses were determined on a Perkin-Elmer 240 C microanalyzer.

Synthesis of Spiro[polycyclic-2'-[1',3']oxathialan]-5'-one Derivatives (2a–d); *General Procedure*

Each compound **1a–d** (0.01 mol) was dissolved in 100 mL of dry toluene, and to this solution 0.01 g of *p*-toluenesulfonic acid was added. Then the reaction mixture was refluxed, whereby the calculated volume of liberated water was removed using a water separator. At the completion of water removal, the reaction mixture was concentrated to 50 mL by distillation and cooled to room temperature, whereby compounds **2a–d** were precipitated, filtered off, and dried. Yields, melting points, and spectral analyses are depicted in Table 1.

Synthesis of Spiro[polycyclic-1'-isothiochroman]-4'-one Derivatives (3a–t); *General Procedure*

Each compound **2a–d** (0.001 mol) was dissolved in 25 mL of the appropriate arene and, to this solution, 0.007 mol of anhydrous aluminum chloride was added. Then the reaction mixture was refluxed for 12 hours. At the end of the reflux time, the reaction mixture was cooled to room temperature, poured into 100 mL of cold 10% hydrochloric acid solution and extracted with chloroform. The extract was washed with 10% sodium carbonate solution, then with water, and dried over anhydrous magnesium sulfate. The solvent was removed by distillation to afford the target products **3a–t**. Results are shown in Table 1.

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