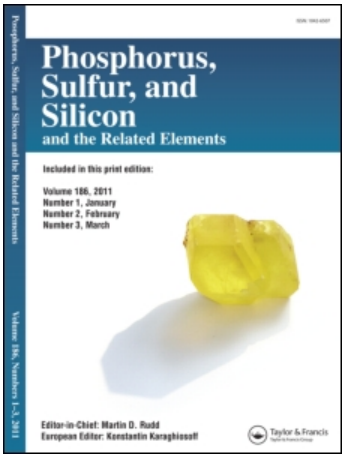

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Synthesis and Absorption Properties of Some 4-Thioaryl-1,8-naphthalimides and 4-Thioaryl-7*H*-benzimidazo-[2,1-*a*]-benz-[*d,c*]-isoquinolin-7-one Derivatives

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A series of novel 4-thioaryl-1,8-naphthalimide derivatives were synthesized using 4-chloro-1,8-naphthalic anhydrid and arylthioles as starting materials. 4-thioaryl-1,8-naphthalic anhydride was treated with different primary amines and o*-phenylene diamine or 1,2-diaminoethene to produce 4-thioaryl-1,8-naphthalimides and 4-thioaryl-7* H*-benzimidazo-[2,1-*a*]-benz-[*d,c*]-isoquinolin-7-one derivatives, respectively. The UV/VIS absorption properties are discussed.*

Keywords 1,2-Diaminoethene; 4-chloro-1,8-naphthalic anhydride; arylthiol; o-phenylene diamine

INTRODUCTION

Derivatives of 4-substituted-1,8-naphthalimides have found application in a number of areas, including fluorescent dyes,[1–12] laser active media,[13,14] liquid crystal displays,[15] analgetics in medicine,[16–18] fluorescent markers in biology,[19] and light emitting diodes.[20–22] The use of 1,8-naphthalimid derivatives in electronic devices has become increasingly popular due to the good light stability and high quantum yield of suitably substituted derivatives.

In this article, we present the synthesis and the UV/VIS absorption properties of a number of 4-thioaryl-1,8-naphthalimides and 4-thioaryl-7*H*-benzimidazo[2.1-*a*]-benz-[*d,c*]-isoquinolin-7-one derivatives.

RESULTS AND DISCUSSION

In continuation of our ongoing research to develop the synthesis of 1,8-naphthalimides[23] with the aim to prepare 4-thioaryl-1,8-naphthalimide

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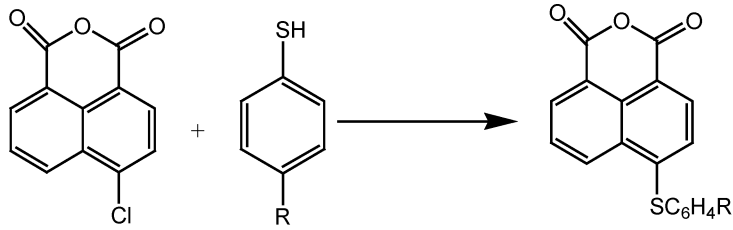
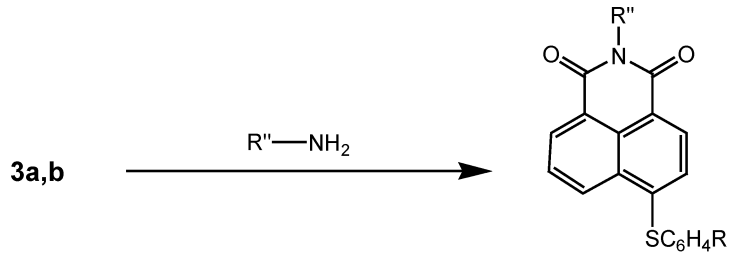
derivatives, we reacted 4-chloro-1,8-naphthalic anhydride **1** with thiophenoles **2a**,**b** in the presence of sodium hydrogen carbonate in boiling DMF[24] (Scheme 1). After the completion of the reaction, the solution was acidified, and the solid products **3a**,**b** were filtered off. For the preparation of the different imides and imidazols, 4-thioaryl-1,8-naphthalic anhydride was reacted with the corresponding amines and 1,2-diamines (Schemes 2–4).



1 **2a,b** **3a,b**

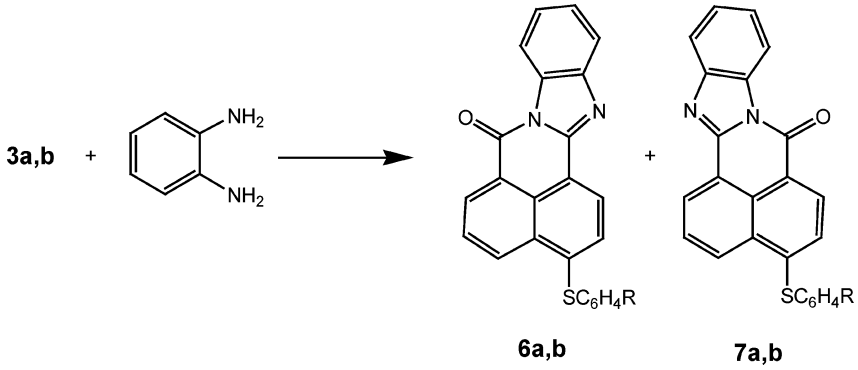
	R
2a, 3a	H
2b, 3b	CH_3

SCHEME 1

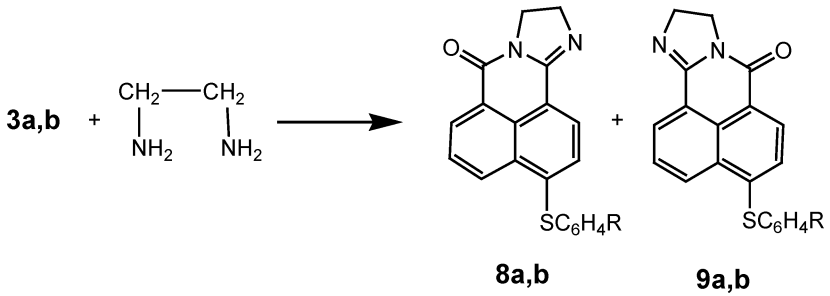


4a–i R=H
5a–i R=CH_3

SCHEME 2



SCHEME 3



SCHEME 4

EXPERIMENTAL

Melting points were determined with an electrothermal 9100 apparatus and are uncorrected. UV spectra were recorded on a UNICAM 8700 series UV/VIS spectrometer. The FTIR spectra were obtained on a Philips 9800 Fourier transform infrared spectrometer. 4-chloro-1,8-naphthalic anhydride was obtained from Aldrich; the other chemicals were obtained from Merck and were used without further purification.

TABLE I Spectroscopic Data and Physical Properties of 1,8-Naphthalic Anhydride and Its Derivatives

Compound	λ_{max} nm	M.P.°C	Yield (%)	Color
1,8-naphtalic anhydride	330.2	256–267	—	Pale yellow
1	338.2	216–217	—	White
3a	386.9	159–160	90	Dark yellow
3b	390.2	179–180	93	Dark yellow

TABLE II Spectroscopic Data and Physical Properties of 4a–i

Compound	R″	M.P.°C	Yield (%)	λ_{max} nm	log ε	Color
4a	$PhCH_2$	188–189	86	395	4.08	Yellow
4b	p-$NO_2C_6H_4$	178–179	76	395.2	4.11	Yellow
4c	o-$NO_2C_6H_4$	186–187	75	394.5	4.12	Yellow
4d	p-$CH_3C_6H_4$	177–178	83	394.4	4.10	Yellow
4e	Ph	192–193	88	392	4.10	Pale yellow
4f	p-$NH_2C_6H_4$	241–243	81	394	4.11	Greenish yellow
4g	p-OHC_6H_4	181–182	82	394.5	4.09	Greenish yellow
4h	p-HO_2C-C_6H_4	194–196	84	393.9	4.14	Bright yellow
4i	p-CH_3-$CONH$-C_6H_4	182–183	87	397.6	4.12	Greenish yellow

The Synthesis of 4-Thiophenyl-1,8-naphtalic Anhydride (3a) and 4-(4-Methylphenyl) thio-1,8-naphthalic Anhydride (3b)

2.32 g (10 mmol) of 4-chloro-1,8-naphtalic anhydride, 15 mmol of the thiophenol, and 0.84 g (10 mmol) of sodium bicarbonate were refluxed in 30 mL of DMF for 7 h. The progress of the reaction was monitored by TLC. The solution was cooled down to an ambient temperature and acidified with 5% aqueous HCl to form a yellow precipitate, which was filtered and recrystallized from ethanol. For spectroscopic data and physical properties, see Table I.

The Synthesis of 4-Thioaryl-*N*-substituted 1,8-Naphthalimides 4a–i and 5a–i

0.01 mol of 4-thioaryl-1,8-naphthalic anhydride and 0.01 mol of the amine was dissolved in 20 mL of 2-methoxyethanol, and the solution was refluxed for 5 h. The progress of the reaction was monitored by TLC. After cooling to r.t. the liquor was poured into 50 mL of distilled water to

TABLE III Spectroscopic Data and Physical Properties of 5a–i

Compound	R″	M.P.°C	Yield (%)	λ_{max} nm	log ε	Color
5a	$PhCH_2$	152–153	81	391.2	4.08	Yellow
5b	p-$O_2NC_6H_4$	175–177	84	391.4	4.13	Yellow
5c	p-$H_3CC_6H_4$	199–200	85	389.8	4.12	Yellow
5d	Ph	202–204	78	389.6	4.04	Pale yellow
5e	p-$H_2NC_6H_4$	162–163	78	388.8	4.11	Greenish yellow
5f	p-HOC_6H_4	262–263	87	390.4	4.13	Yellow
5g	p-$HO_2CC_6H_4$	256–257	85	396.6	4.13	Bright yellow
5h	p-CH_3CONH-C_6H_4	240–243	87	397.2	4.14	Greenish yellow
5i	H_2N	196–197	95	397	4.14	Bright yellow

TABLE IV Spectroscopic Data and Physical Properties of Compounds 6–9

Compounds	R	M.P.°C	Yield (%)	λ_{Max} nm	log ε	Color
6a, 7a	H	196–197	93	418	4.15	Orange
8a, 9a	H	193–194	85	438.1	4.14	Orange
6b, 7b	Me	192–193	91	414.9	4.11	Orange
8b, 9b	Me	194–195	81	441.9	4.09	Orange

form a yellow precipitate. The resulting precipitate was filtered off, and the crude product was recrystallized from ethanol. For spectroscopic data and physical properties, see Tables II and III.

The Synthesis of 4-Thioaryl-7*H*-benzimidazo[1,2-*a*]-benz-[*d,c*]-isoquinolin-7-one Derivatives 6–9

0.01 mol of 4-thioaryl-1,8-naphthalic anhydride was reacted with 0.02 mol of 1,2-diamine in 10 mL of boiling acetic acid for 3 h. After cooling to r.t., 100 mL of distilled water was added to the solution, and the resulting precipitate was filtered off. Recrystallization of the crude product from DMF gave the final products **6–9**. For spectroscopic data and physical properties, see Table IV.

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