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Synthesis and characterization of 3-aryl[1,2,3,5]tetrazino[5,4-*a*]benzimidazole-4(*3H*)-thione derivatives

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ABSTRACT

A series of 3-aryl[1,2,3,5]tetrazino[5,4-*a*]benzimidazole-4(*3H*)-thione derivatives have been synthesized. The compounds were synthesized in excellent yields (65-75%) and the structures were established on the basis of corresponding IR, ^{1}H NMR, Mass and elemental analysis data. The purity has been ascertained on the basis of chromatographic resolution using CH_2Cl_2 as eluent.

Keywords: *3-aryl[1,2,3,5]tetrazino[5,4-a]benzimidazole-4(3H)-thione, Synthesis, Derivatives, Medicinal use, Energetic*

Introduction

Recently there has been considerable interest in the study of the reactivity and properties of six-membered tetrazine heterocycles [1-14]. The high degree of attention given to this heterocyclic system stems from the unique and interesting properties displayed by six-membered tetrazines. Tetrazines have demonstrated powerful synthetic utility through their ability to participate in inverse electron demand Diels-Alder reactions, providing access to a wide range of other heterocycles and natural products. These materials are of use in the area of energetic chemistry. This electroactive, colored ring system typically exhibits high electron affinity, low lying π* orbitals and n-π* transitions in the visible light region, attractive properties for optical and electroactive material applications. Furthermore, tetrazines also possess high positive heats of formation and crystal densities, properties important in energetic chemistry. The chemistry of tetrazines has gained increased attention in the last few decades, due mostly to their applications in organic synthesis [15,16], crop protection [17,18], pyrotechnics (high nitrogen content energetic materials [19-20]) etc. Very recently, research to obtain new compounds for their optical and electrochemical properties has become increasingly active because these compounds have a huge potential especially in sensor applications [21-24].

The interesting characteristics features found in many of these tetrazines are their potential biological activity as antiproliferative and antitumorous compounds. *s*-Tetrazine derivative including hexahydro, 1,6-dihydro, 1,4-dihydro-, 1,2-dihydro- and aromatic *s*-tetrazine exhibit antitumorous activities with 50% inhibition rate against P-388 cell and A-549 cancer cell growth [25]. Azolotetrazinones have been the focus of medicinal chemists in the past decades because of the outstanding antineoplastic activity exhibited by them. Likewise, mitozolomide [26] and temozolomide [27] have attracted remarkable attention owing to their efficiency against malignant melanoma, mycosis fungoides, and brain tumors. Therefore, it is revealed that a tetrazine ring can be an effective pharmacophore in various types of medicinal activities.

Most of the recent reports on the chemistry of tetrazines have focused on the development of methods for preparing difficult-to-access nonsymmetrically substituted tetrazines. The majority of successful methods use soft, neutral heteroatom nucleophiles for the displacement of leaving groups at the 3 and 6 positions of the tetrazine. The use of hard carbanion nucleophiles results

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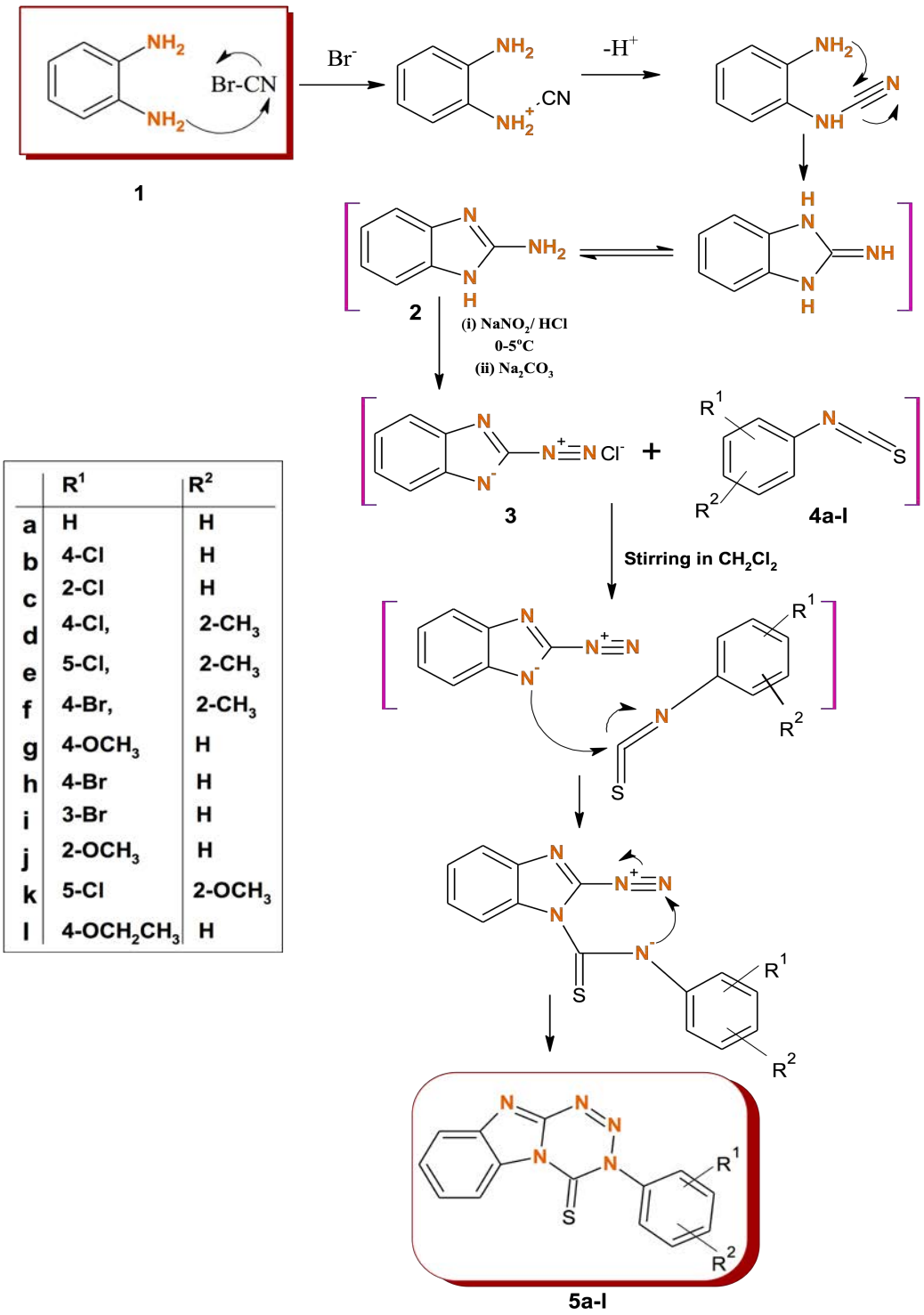
in unusual azaphilic addition to the tetrazine core. Only a few reports have documented the reactivity of tetrazines toward anionic heteroatom nucleophiles. In these cases, the reported yields are typically low and undesired side reactions are generally observed. Several routes were proposed to synthesize tetrazine derivatives including the reactions of hydrazine with nitriles [28], imino esters [29], thioamides [30], dithioamides, aroyl chloride azines [31] etc.

Thus, buoyed from these aforementioned medicinal use and synthetic challenges associated with the six-membered tetrazines, tailoring of some new pharmacologically significant tetrazine compounds was endeavored for the sake of present investigation.



	R^1	R^2
a	H	H
b	4-Cl	H
c	2-Cl	H
d	4-Cl,	2-CH_3
e	5-Cl,	2-CH_3
f	4-Br,	2-CH_3
g	4-OCH_3	H
h	4-Br	H
i	3-Br	H
j	2-OCH_3	H
k	5-Cl	2-OCH_3
l	4-OCH_2CH_3	H

Scheme I. Synthetic pathway for 3-aryl[1,2,3,5]tetrazino[5,4-*a*]benzimidazole-4(3*H*)-thione derivatives (5a-l)

Table 1: Physical characteristic data for 3-aryl[1,2,3,5]tetrazino[5,4-*a*] benzimidazole-4(*3H*)-thiones (5a-l)

S. No.	Comp	M.P. (°C)*	Yield (%)	Analytical data Found (Calcd.) (%)		
				C (%)	H (%)	N (%)
1.	5a	135-137	72	60.14 (60.20)	3.17 (3.25)	**24.96(25.07)**
2.	5b	101-103	65	53.49 (53.59)	2.51 (2.57)	**22.26(22.32)**
3.	5c	122-123	70	53.52 (53.59)	2.52 (2.57)	**22.25(22.32)**
4.	5d	137-139	69	54.89 (54.96)	3.01 (3.07)	**21.28(21.37)**
5.	5e	120-122	73	54.91 (54.96)	3.03 (3.07)	**21.32(21.37)**
6.	5f	114-115	71	48.35 (48.40)	2.68 (2.71)	**18.76(18.81)**
7.	5g	131-132	75	58.19 (58.24)	3.51 (3.58)	**22.57(22.64)**
8.	5h	126-128	72	46.88 (46.94)	2.19 (2.25)	**19.48(19.55)**
9.	5i	133-135	71	46.89 (46.94)	2.21 (2.25)	**19.51(19.55)**
10.	5j	114-116	70	58.20 (58.24)	3.52 (3.58)	**22.59(22.64)**
11.	5k	101-103	69	59.33 (59.43)	4.96 (4.05)	**21.59(21.66)**
12.	**5l**	**142-143**	**65**	**59.71 (59.80)**	**3.42 (3.45)**	**22.63(21.79)**

Materials and Methods

Chemistry

Herein we report a benign and convenient synthesis of 3-aryl[1,2,3,5]tetrazino[5,4-*a*]benzimidazole-4(*3H*)-thiones derivatives starting from initial reaction of *o*-phenylene diamine **1** with cyanogen bromide. The treatment was performed in aqueous medium in the presence of NaOH. A dark oily layer separated on cooling and crystallized on standing to afford buff white crystals of 2-amino benzimidazole **2,** which further undergoes diazotization at 0 °C to afford azo derivative of 2-amino benzimidazole. Then the same solution was neutralized via saturated aqueous Na_2CO_3 to allow the formation of zwitter ion. These zwitter ions were further treated with aryl isothiocyanate derivatives in dichloromethane and stirred for 48 hours to afford the desired products in good yield. After completion of reaction as monitored by TLC, the solvent was removed in vacuum. The crude product was purified by column chromatography using CH_2Cl_2 as eluent to afford **5a-l** in 65-75% yield. An overview of synthetic pathway is delineated in Scheme-I and physical characteristics of all the synthesized compounds are given in Table 1.

Experimental Details

All the chemicals used were of AR grade purity. IR spectra were recorded on Perkin Elmer model 377 spectrophotometer in KBr pellets. IR frequencies were measured in cm^{-1}. ^1H NMR spectra were recorded in $CDCl_3$ solution in 5 mm tubes at 25 °C on a Bruker DRX-300 instrument (300 MHz FT NMR with low and high temperature facility -90 °C to +80 °C) with deuterium signal as the lock and TMS as internal standard. Chemical shifts were measured in ppm units. The FAB mass spectra were recorded on a JEOL SX102/DA–6000 Mass Spectrometer using Argon/Xenon (6 kv, 10 mA) as the FAB gas. The ESI mass spectra were recorded on a Micromass Quattro II triple quadrapole mass spectrometer. The melting points were determined on an electric melting point apparatus (Biotech Bombay, India) in open capillaries and are uncorrected. The purity of all the synthesized compounds was ascertained by TLC resolution on silica gel-G (E Merck). Elemental analyses were performed on Elemental Vario, EL III Carlo Erba 1108.

1. Synthesis of 3-Aryl[1,2,3,5]tetrazino[5,4-*a*]benzimidazole-4(*3H*)-thiones (5a-l)

The derivatives were synthesized in the following sequential steps-

Step [A] : Synthesis of *1H*-benzimidazol-2-amine (2)

In a round-bottomed flask cyanogen bromide (7.0 gm, 0.067 mol) was added in small portions, with shaking to a suspension of *o*-phenylene diamine (7.2 gm, 0.067 mol) in 80 ml water, considering the directions set for the preparations of benzimidazole. The solution was filtered after standing

overnight. Then sodium hydroxide (2.68 gm, 0.067 mol) dissolved in 6.0 ml water was added and the solution was evaporated on a steam bath. A dark oily layer separated on cooling and crystallized on standing to afford buff white crystals of *1H*-benzimidazol-2-amine **2** in good yield.

IR (, cm^{-1}): 3460 (NH asym.), 3410 (NH sym.), 3070 (C-H, sp^2), 1658 (C=N), 1605, 1590, 1420 (C⎓C, ring str.), 910, 870, 740 (sub. phenyl); ^{1}HNMR (ppm) : 4.8 (s, 2H, NH_2), 6.1 (s, 1H, NH), 7.14-7.28 (s, 4H, C_6H_4); FAB-MS m/z: M^++1 [134 (11%)]; Yield 81 %; m.p. 229-230 °C; Anal. Calcd for $C_7H_7N_3$: C, 63.14; H, 5.30; N, 31.56. Found: C, 63.11; H, 5.28; N, 31.51.

Step [B] : Synthesis of aryl isothiocynate derivatives (4a-l)

In a round-bottomed flask, placed in ice salt mixture and equipped with a mechanical stirrer and a separatory funnel, 45 ml of concentrated ammonia and 37 ml of pure CS_2 was introduced slowly by maintaining the temperature 0-5 °C. The mixture was stirred and the various pertinent aniline derivatives (0.30 mol) were added slowly with stirring for further 30 minutes. Then the mixture was allowed to stand for half an hour. A heavy precipitate of dithiocarbamate separates. The salt was extracted by four extractions with 100 ml water and to the resulting solution 100 gm of lead nitrate in 200 ml of water was added to precipitate lead sulphide. This solution was steam distilled in to a receiver containing 5 ml of 0.5 M sulphuric acid. The organic layer was separated and dried and distilled under reduced pressure to afford the aryl isothiocyanate derivatives **4a-l**. Some derivatives (**4a**, **4b**, **4c**, **4h**, **4i**, **4l**) were synthesized using respective aniline derivatives by above procedure and remaining were procured.

Step [C]: Synthesis of 3-aryl[1,2,3,5]tetrazino[5,4-*a*]benzimidazole-4(*3H*)-thiones (5a-l)

In a beaker *1H*-benzimidazol-2-amine **2** (0.36 gm, 0.003 mol) was dissolved in conc. hydrochloric acid. In another beaker sodium nitrite (0.21 gm, 0.003 mol) was taken in 2.0 ml water. After cooling, the sodium nitrite solution was added to the solution of *1H*-benzimidazol-2-amine.HCl by maintaining the temperature 0-5 °C to diazotize the amine. Then this solution was neutralized via saturated aqueous Na_2CO_3 (1.27 gm, 0.012 mol) to allow the formation of zwitter ion. Then, aryl isothiocyanate (0.002 mol) in 5ml of dichloromethane was added and mixture was stirred for 48 hours at room temperature. After completion of reaction as monitored by TLC, the solvent was removed in vacuum. The crude product was purified by column chromatography using CH_2Cl_2 as eluent to afford **5a-l** in good yields. The spectroanalytical data's of some representative compounds are given below:

a. 3-Phenyl[1,2,3,5]tetrazino[5,4-*a*]benzimidazole-4(*3H*)-thione (5a):

Prepared according to the general procedure as in experimental section.

IR (, cm^{-1}): 3000 (C-H, sp^2), 1623 (C=C/ C=N),1578(-N=N), 1530, 1471,1375 (C-H bending), 1250 (-C=S), 1074(C-N str.), 945, 892, 824 (sub phenyl); ^{1}HNMR (ppm) : 7.10-7.34 (m, 9H, C_6H_5); Mass [m/z (% RA)]: M^+ [279.2 (20 %)]

b. 3-(4-Chlorophenyl)[1,2,3,5]tetrazino[5,4-*a*]benzimidazole-4(*3H*)-thione (5b):

Prepared according to the general procedure as in experimental section.

IR (, cm^{-1}): 3030 (C-H, sp^2), 1631 (C=C/C=N), 1551 (-N=N), 1525, 1470,1374 (C-H bending), 1251 (-C=S),1071(C-N str.), 941, 890, 814 (sub phenyl), 661 (C-Cl); ^{1}HNMR (ppm) : 6.97-6.99 (m, 4H, C_6H_4) , 7.51-7.53 (dd, 2H, Ar-Ha), 7.58-7.60 (dd, 2H, Ar-Hb); Mass [m/z (% RA)]: 313 (M^+, 21), 278 (M-Cl, 100), 202 (24), 77 (51).

c. 3-(4-Bromo-2-methylphenyl)[1,2,3,5]tetrazino[5,4-*a*]benzimidazole-4(*3H*)-thione (5f):

Prepared according to the general procedure as in experimental section.

IR (, cm^{-1}): 3049 (C-H, sp^2), 2981, 2880 (C-H, asym., sym.), 1642 (C=C/C=N), 1598 (-N=N), 1521 1460,1414 (C-H bending), 1263 (-C=S),1113 (C-N str.), 899, 860, 774 (sub phenyl), 596, 530 (C-Br); ^{1}HNMR (ppm) : 2.31 (s, 3H, $-CH_3$), 7.21-7.51 (m, 7H, C_6H_4); Mass [m/z (% RA)]:372(M^+, 44), 358 (11), 357 (100), 293 (44), 279 (15), 208 (20), 158 (51), 140 (33).

d. 3-(4-Bromophenyl)[1,2,3,5]tetrazino[5,4-*a*]benzimidazole-4(*3H*)-thione (5h):

Prepared according to the general procedure as in experimental section.

IR (, cm^{-1}): 3021 (C-H, sp^2), 1638 (C=C/C=N), 1557 (-N=N), 1520 1457,1411 (C-H bending), 1254 (-C=S),1107 (C-N str.), 897, 854, 751 (sub phenyl), 581, 524 (C-Br); ^{1}HNMR (ppm) : 6.70-6.91 (m, 4H, C_6H_4) , 7.04-7.06 (dd, 2H, Ar-Ha), 7.26-7.36 (dd, 2H, Ar-Hb); Mass [m/z (% RA)]: 358 (M^+, 14), 279 (M-Br, 100), 202 (31), 77 (56).

Conclusion

Conclusively, a series of 3-aryl[1,2,3,5]tetrazino[5,4-*a*]benzimidazole-4(*3H*)-thione derivatives were synthesized in appreciable yields and characterized from all spectroanalytical methods. These derivatives have a wide number of usages in all the areas of medicinal and energetic chemistry, therefore they can be used as a lead compounds for such kind of studies.

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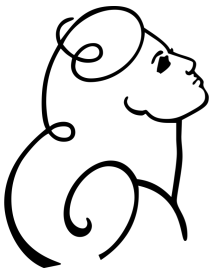

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