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A FACILE PHOTOCHEMICAL SYNTHESIS OF 12H-BENZOTHIAZOLO[2,3-b]QUINAZOLIN-12-ONES

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Abstract: The photochemical synthesis of 12H-benzothiazolo[2,3-b]quinazolin-12-ones **(4)** by the irradiation of 2-thioquinazolinediones **1** and disulphide **5** are described.

We have earlier reported [1-3] the formation of benzothiazole ring system by the irradiation of o-halothioacetanilides. The synthesis of new derivatives of benzothiazoles with a variety of substituents at the 2-position was also developed[4]. In continuation of our efforts to develop the photochemical route for the synthesis of novel heterocyclic systems, we have synthesized the benzothiazolo[2,3-b]quinazolinones by the facile photochemical method which is reported herein.

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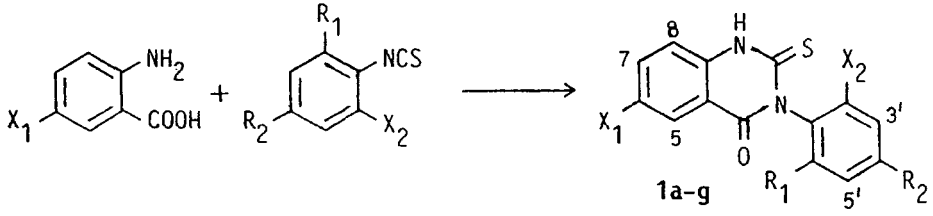
Bose and Pathak[5] synthesized benzothiazoloquinazolinone **4** by the condensation of anthranilic acids or esters with 2-chlorothiazoles. Katz[6], McCarty[7] and later on Lunn and Harper[8] repeated the same reaction and studied the properties of **4**; the drawback of this synthetic approach was the poor yield in the preparation of the 2-chlorobenzothiazole[5]. Condensations of isatoic anhydrides with sodium salt of 2-amino-5-substituted thiophenols followed by treatment with ethyl chloroformate furnished[9] 12H-benzothiazolo [2,3-b]quinazolin-12-ones. The cyclising agent, N-halosuccinimide/sulphuric acid[10] permits the preparation of fused benzothiazoles.

Reaction of anthranilic acid with phenyl isothiocyanate under reflux in ethanol furnished[11] the 3-phenyl-2-thio-2,4(1H,3H)quinazolinedione. By a slight modification of the above procedure (Method A), 3-aryl-2-thioquinazolinediones **1a-e** were prepared. However, preparation of compounds **1f** and **1g** by Method A furnished them as a mixture of the expected quinazolinone and the thiazine product as per IR data, which were not separated. This was circumvented by carring out the addition of the substituted phenyl isothiocyanate to an alcoholic solution of anthranilic

acid at 0°C and allowing the reaction to attain room temperature gradually; refluxing for 4 hours afforded the products **1f** and **1g**. The reaction of anthranilic acid with 2,3-dichlorophenyl- or 1-chloro-2-naphthyl isothiocyanate, however, did not furnish the expected quinazoline ring system by method A or B; instead, only the respective benzothiazine derivatives **2** and **3** were isolated. The benzothiazine ring **2** is known to be obtaind from 2-thioquinazolinedione **1** on treatment with concentrated sulphuric acid[11].



1a-g

1a: X_1=H; X_2=Cl; R_1=H; R_2=H

1b: X_1=H; X_2=Br; R_1=H; R_2=H

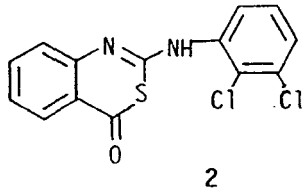
1c: X_1=H; X_2=Br; R_1=H; R_2=CH_3

1d: X_1=H; X_2=Br; R_1=CH_3; R_2=CH_3

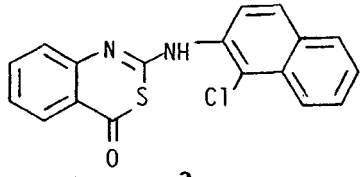
1e: X_1=H; X_2=Cl; R_1=CH_3; R_2=H

1f: X_1=Cl; X_2=Cl; R_1=H; R_2=H

1g: X_1=Cl; X_2=Br; R_1=H; R_2=CH_3



2

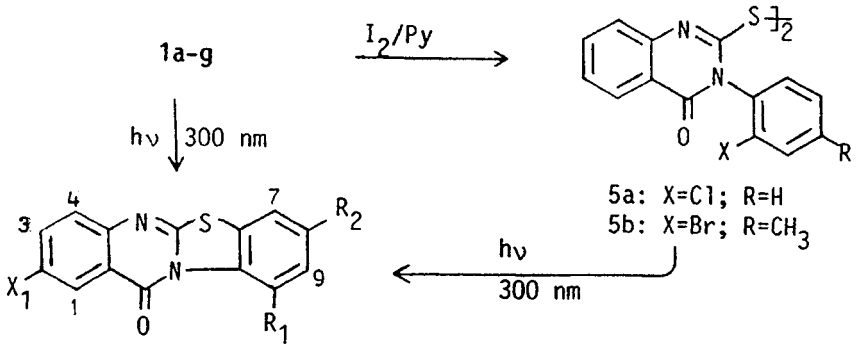


3

The 2-thioquinazolinediones **1a-g** were dissolved in methanol, purged with nitrogen for half an hour and irradiated at 300 nm; chromatographic purification

furnished the respective 12H-benzothiazolo[2,3-b] quinazolinones **4a-g** with moderate to good yields.

It was also of interest to study the photochemistry of the disulphide **5** which was prepared from **1** by oxidation[10] with iodine in aqueous pyridine.



The disulphides **5a** and **5b** were irradiated with 300 nm for 37 and 14 hours in chloroform which furnished **4a** and **4c** in 48 and 66% yields respectively. It is to be noted that a disulphide is one of the products in the photolysis of arylthiol esters[12], xanthates[13] and benzyl toluene-α-thiosulphonate[14].

The IR spectrum of the 2-thioquinazolinediones **1a-g** showed a characteristic absorption around 3240 (NH), and 1670 (C=O) cm^{-1} whereas those of benzothiazine derivatives **2** and **3** appeared in the region 3180-2960 and 1700 (C=O) cm^{-1}. In the ^{1}H-NMR spectra of all the benzothiazoloquinazolinones **4a-g** the

down field shift[10] of H-1 and H-10 are specially noticeable. The mass spectra of the quinazolinediones **1** show the fragments due to the loss of halogen which then apparently loses a hydrogen giving benzothiazole ions. The 6-methyl and 4,6-dimethyl compounds **4d, 4e** show the loss of ·OH from the molecular ion which must arise by the hydrogen transfer from the methyl group to the carbonyl oxygen.

EXPERIMENTAL SECTION

Melting points are uncorrected. IR spectra were recorded on a Perkin-Elmer 258 spectrophotometer on KBr disc. The ^{1}H-NMR spectra were recorded on a Varian EM 390 and Jeol GSX 400 spectrometers. Mass spectra were recorded on Hewlett Packard 5985 and Shimadzu QP 1000A. Irradiations were carried out in a quartz vessel in a Rayonet photochemical reactor model RPR-208 equipped with seven low pressure mercury lamps of wavelength 300 nm. Chromatographic purifications were performed on silica gel (100-200 mesh).

General procedure for the preparation of 3-substituted phenyl-2-thio-2,4(1H,3H)quinazolinedione

Method A: Anthranilic acid (2.0g, 15 mmol) was dissolved in ethyl alcohol (30 ml) and 2-chlorophenyl isothiocyanate (2.5 g, 15 mmol) added dropwise at room temperature with stirring. The reaction mixture was refluxed for one hour, when a crystalline product formed, which was further refluxed for half an hour; the reaction mixture was cooled to room temperature,

filtered, washed with alcohol, dried and crystallised from ethanol.

3-(2-Chlorophenyl)-2-thio-2,4(1H,3H)quinazolinedione (1a): Yield (3.2g)74%, m.p. 246-248°C. IR:3200 (NH), 1660 (C=O), 1610, 1520, 1400, 1195 cm^{1}. UV(MeOH): λmax(ε) 294(30,960) and 226(40,660)nm. ^{1}H-NMR(DMSO-d_6):δ 7.31-7.41 (m, 2H, ArH), 7.45-7.52 (m, 3H, ArH) 7.56-7.60 (m, 1H, ArH), 7.73-7.79 (m, 1H, ArH), 8.01 (dd, J = 8Hz, J'= 2Hz, 1H, H-5), 13.14 (br s, 1H, NH). MS, m/z(%):253 (M^{+}-Cl, 100), 224(5), 150(5), (126(9), 92(8), 90(9). Anal. Calcd. for $C_{14}H_9ClN_2OS$: C, 58.23; H, 3.14; N, 9.70. Found: C, 58.61; H, 2.98, N, 9.69.

3-(2-Bromophenyl)-2-thio-2,4(1H,3H)quinazolinedione (1b): Reaction time 2 hours, yield 76%, m.p. 256-258°C (lit[15] m.p. 266-268°C). IR:3200, 1660, 1610, 1510, 1390, 1250 cm^{1}. UV(MeOH): λmax 294 and 222nm. ^{1}H-NMR (DMSO-d_6):δ 7.31-7.91 (m,7H, ArH),7.9-8.0 (m, 1H, H-5), 13.2 (br s, 1H, NH), MS, m/z(%):253(M^{+}-Br, 100), 224(13), 167(15), 108(11), 92(30), 90(23).

3-(2-Bromo-4-methylphenyl)-2-thio-2,4(1H,3H)quinazolinedione (1c): Reaction time 1.5 hours, yield 86%, m.p. 215-217°C. IR:3260, 1665, 1620, 1530, 1400, 1200 cm^{-1}. UV(MeOH): λmax(ε) 295(27,100) and 226(36,710)nm.

^{1}H-NMR (DMSO-d_6):δ 2.40 (s, 3H, CH_3), 7.30-7.39 (m, 3H, ArH), 7.48 (d,J = 8.8Hz, 1H, ArH), 7.57 (s, 1H, ArH), 7.78 (t, 1H, ArH), 7.98 (d,J = 8.7Hz, 1H, H-5), 13.12 (s, 1H, NH) MS, m/z(%): 267(M^+-Br, 100), 266(12), 237(5), 224(4), 133(25), 119(13), 92(7), 90(12). Anal. Calcd. for $C_{15}H_{11}BrN_2OS$: C, 51.87; H, 3.19; N, 8.07. Found: C, 51.91; H, 3.46; N, 7.93.

3-(2-Bromo-4,6-dimethylphenyl)-2-thio-2,4(1H-3H)quinazolinedione (1d): Reaction time 2 hours, yield 69%, m.p. 271-273°C. IR:3260, 1670, 1620, 1530, 1485, 1390, 1195 cm^{-1}. UV(MeOH): λmax 295 and 222 nm. ^{1}H-NMR(DMSO-d_6) δ 2.12 (s, 3H, CH_3), 2.36(s, 3H, CH_3), 7.17 (s, 1H, H-5'), 7.37 (t, J = 7.9Hz, 1H, H-6), 7.40 (s, 1H, H-3'), 7.50 (d, J = 8.2Hz, 1H, H-8), 7.80 (t, J = 8.0Hz, 1H, H-7), 8.01 (dd, J = 8.2Hz, J' = 2Hz, 1H, H-5), 13.26 (s, 1H, NH). MS, m/z(%): 281(M^+-Br,100), 261(10), 256(10), 220(48), 205(23), 189(13), 141(15), 133(13), 98(15), 90(15). Anal. Calcd. for $C_{16}H_{13}BrN_2OS$: C, 53.20; H, 3.63; N,7.75. Found:C, 53.24; H,3.71; N,7.64.

3-(2-Chloro-6-methylphenyl)-2-thio-2,4(1H,3H)quinazolindione (1e): Reaction time 3 hours, yield 66%, m.p. 267-269°C. IR:3240, 1655, 1620, 1530, 1485, 1390, 1200 cm^{-1}. UV(MeOH): λmax 294 and 221 nm.^{1}H-NMR(DMSO-d_6): δ 2.18 (s, 3H, CH_3), 7.28-7.42 (m, 4H, ArH), 7.51 (d, J =

8.3Hz, 1H, ArH), 7.75 (t, J = 8.1Hz, 1H, ArH), 8.0 (d, J = 8.8Hz, 1H, H-5), 13.17 (br s, 1H, NH). Anal.Calcd. for $C_{15}H_{11}ClN_2OS$: C, 59.50; H, 3.66; N, 9.25. Found:C, 59.38; H, 3.75; N, 9.33.

Method B: 2-Chlorophenyl isothiocyanate (1.7g, 10 mmol) was added dropwise with stirring to an alcoholic solution (15 ml) of 5-chloroanthranilic acid[16] (1.7g, 10 mmol) at 0°C for one hour and the reaction mixture allowed to attain room temperature gradually. The reaction mixture was further refluxed for 4 hours and cooled to isolate the product which was filtered, washed with alcohol, dried and crystallised from ethanol.

6-Chloro-3-(2-chlorophenyl)-2-thio-2,4(1H,3H)quinazolinedione (1f): Reaction time 4 hours, yield (2.05g) 64%, m.p. 261-263°C. IR:3250, 1670, 1615, 1520, 1480, 1380, 1210 cm^{-1}. UV(MeOH): λmax 299 and 217 nm. ^{1}H-NMR(DMSO-d_6): δ 7.32-7.36 (m, 1H, ArH), 7.44-7.48 (m, 2H, ArH), 7.50 (d, J = 8.7Hz, 1H, ArH), 7.55-7.59 (m, 1H, ArH), 7.70 (dd, J = 8.7Hz, J' = 2Hz, 1H, ArH), 7.97 (d, J = 2Hz, 1H, H-5), 13.20 (br s, 1H, NH). Anal. Calcd. for $C_{14}H_8Cl_2N_2OS$: C, 52.03; H, 2.50; N, 8.67. Found: C, 52.29; H, 2.61; N, 8.62.

6-Chloro-3-(2-bromo-4-methylphenyl)-2-thio-2,4(1H,3H)-quinazolinedione (1g): Reaction time 4 hours, yield 66% m.p. 258-260°C. IR:3240, 1680, 1610, 1515, 1390, 1210 cm^{-1}. UV(MeOH): λmax 299 and 223 nm. ^{1}H-NMR(DMSO-d_6):δ 2.42 (s, 3H, CH_3), 7.22 (d, J = 7.8Hz, 1H, H-6'), 7.30 (d, J = 7.3Hz, 1H, H-5'), 7.49 (d, J = 8.7Hz, 1H, H-8), 7.50 (br s, 1H, H-3'), 7.69 (dd, J = 8.7Hz, J' = 2.1Hz, 1H, H-7), 7.94 (d, J = 2.1 Hz, 1H, H-5), 13.23 (br s, 1H, NH). Anal. Calcd. for $C_{15}H_{10}BrClN_2OS$: C, 47.19; H, 2.64; N, 7.34. Found: C, 47.28; H, 2.49; N, 7.48.

2,2'-Dithiobis[3-(2-Chlorophenyl)-4(3H)-quinazolinone] (5a): To a vigorously stirred solution of the compound **1a** (580 mg, 2mmol) in water/pyridine (25/10ml), a solution of iodine (254 mg, 1 mmol) in ethyl alcohol (20 ml) was added dropwise at room temperature. The mixture was stirred for 30 minutes and the resulting solid filtered washed with water and dried. Yield (425 mg) 74%, m.p. 253-255°C. IR:1685, 1575, 1550, 1465, 1265, 765 cm^{-1}. ^{1}H-NMR($CDCl_3$):δ 7.5-8.0 (m, 7H, ArH), 8.52 (d, 1H, J = 9Hz, H-5).

2,2'-Dithiobis[3-(2-bromo-4-methylphenyl)-4(3H)-quinazolinone] (5b) was prepared likewise from **1c** in 87% yield, m.p. 190-192°C. IR: 1685, 1570, 1550, 1460, 1245, 1190, 760 cm^{-1}. ^{1}H-NMR($CDCl_3$):δ 2.50 (s, 3H,

CH_3), 7.50-8.03 (m, 6H, ArH), 8.49 (d, 1H, J = 9Hz, H-5).

2-(2,3-Dichlorophenylamino)-4H-3,1-benzothiazin-4-one (2). Reaction of 2,3-dichlorophenyl isothiocyanate (2.0g, 10mmol) and anthranilic acid (1.37g, 10 mmol) carried out by method A or Bafforded the product **2**. Yield (2.82g) 88%, m.p. 291-293°C, IR: 3180, 3120, 3020, 2970, 1710, 1620, 1540, 1210 cm^{-1}. ^{1}H-NMR(DMSO-d_6:δ 7.03-7.80 (m, 7H, ArH), 11.36 (br s, 1H, NH). Anal. Calcd. for $C_{14}H_8Cl_2N_2OS$: C, 52.03; H, 2.50; N, 8.67. Found: C, 52.24; H, 2.56; N, 8.63.

2-(1-Chloro-2-naphthalenylamino)-4H-3,1-benzothiazin-4-one (3) Reaction of 1-chloro-2-naphthyl isothiocyanate (2.2g, 10 mmol) and anthranilic acid (1.37g, 10mmol) carried out by method A or B afforded the product **3**. Yield (2.3g) 68%, m.p. 287-289°C. IR: 3170, 3100, 3010, 2960, 1700, 1630, 1535, 1410, 1200 cm^{-1}. ^{1}H-NMR(DMSO-d_6):δ 7.0-8.03 (m, 10H, ArH), 12.7 (br s, 1H, NH). Anal. Calcd. for $C_{18}H_{11}ClN_2OS$: C, 63.81; H, 3.27; N, 8.27. Found: C, 63.29; H, 3.08; N, 8.60.

General procedure for the preparation of benzo thiazolo[2,3-b]quinazolin-12-ones. (4a-g from 1a-g) Generally the 2-thioquinazolinedione was not easily

soluble in methanol. The well powdered respective 2-thioquinazolinedione (3 mmol) was dissolved in methanol (200ml) and purged with nitrogen gas for half an hour. The solution was irradiated at 300 nm in a quartz vessel. The reaction was followed by TLC and continued until the disappearance of the starting material. Methanol was distilled off under reduced pressure when a solid separated out which was purified by passing through a column of silica gel; elution with benzene furnished the respective benzothiazolo[2,3-b] quinazolin-12-one which was crystallised from benzene or chloroform. No other product was isolated from column chromatography.

12H-Benzothiazolo[2,3-b]quinazolin-12-one(4a)

The irradiation of 2-thioquinazolindione **1a** at 300 nm for 45 hours afforded the product **4a**. The 2-thioquinazolindione **1b** was irradiated at 300 nm for 21 hours which afforded the product **4a**. Yield 51% (from **1a**), 72% (from **1b**); m.p. 190-192°C (lit[17] m.p. 193°C). IR:1685 (C=O), 1590, 1550, 1470, 1250, 760 cm^{-1}. ^{1}H-NMR ($CDCl_3$):δ 7.13-7.60 (m, 6H, ArH), 8.20 (dd, 1H, J = 8Hz, J' = 2Hz, H-1), 8.76-8.86 (m, 1H, H-10). MS, m/z(%): 252(M^+, 100), 224(20), 223(9), 134(6), 126(7), 112(17), 90(10).

8-Methyl-12H-benzothiazolo[2,3-b]quinazolin-12-one(4c): Irradiation time 15 hours, yield 65%, m.p. 230-232°C (lit[5] m/p.226°C). IR: 1690, 1590, 1550, 1470, 770 cm^{-1}. ^{1}H-NMR($CDCl_3$):δ 2.41 (s, 3H, CH_3), 7.22-7.48 (m, 5H, ArH), 8.40 (dd, 1H, J = 8Hz, J' = 2Hz, H-1), 8.84 (d, 1H, J = 8Hz, H-10). ^{13}C-NMR($CDCl_3$): 21.4 (q, CH_3), 118.7, 119.0, 122.0, 123.7, 125.8, 126.0, 127.2, 127.8, 134.8, 137.0, 147.4, 210.0. MS, m/z(%): 266(M^+, 100), 265(21), 238(6), 237(17), 133(14), 119(8), 90(6).

8,10-Dimethyl-12H-benzothiazolo[2,3-b]quinazolin-12-one (4d): Irradiation time 12 hours, yield 48%, m.p. 189-191°C, IR: 1705, 1580, 1550, 1470, 770 cm^{-1}. ^{1}H-NMR ($CDCl_3$):δ 2.36 (s, 3H, CH_3), 2.56 (s, 3H, CH_3), 7.0-7.70 (m, 5H, ArH), 8.23 (dd, 1H, J = 8Hz, J' = 2Hz, H-1). MS, m/z(%): 280(M^+, 100), 265(15), 263(21), 116(13), 102(15), 90(3). Anal. Calcd. for $C_{16}H_{12}N_2OS$:C, 68.30; H, 2.81; N, 9.96. Found: C, 67.8; H, 3.01; N, 10.32.

10-Methyl-12H-benzothiazolo[2,3-b]quinazolin-12-one (4e): Irradiation time 30 hours, yield 42%, m.p. 153-155°c. IR:1710, 1580, 1550, 1465, 760 cm^{-1}. ^{1}H-NMR ($CDCl_3$):δ 2.6 (s, 3H, CH_3), 7.29-7.37 (m, 2H, ArH), 7.41-7.48 (m, 2H, ArH), 7.63 (d, J = 8.3Hz, 1H, ArH), 7.76-7.80 (m, 1H, ArH), 8.33 (dd, 1H, J = 8.3Hz, J' =

1.5 Hz, H1). MS, m/z(%):266(M^+, 100), 249(30), 237(19), 121(23), 102(32), 90(34). Anal. Calcd. for $C_{15}H_{10}N_2OS$: C, 67.65; H, 3.78; N, 10.52. Found: C, 67.74; H, 3.87; N, 10.42.

2-Chloro-12H-benzothiazolo[2,3-b]quinazolin-12-one (4f): Irradiation time 33 hours, yield 64%, m.p. 222-224°C (lit[10] m.p. 225-226°C). IR:1690, 1580, 1560, 1540, 1460, 755 cm^{-1}. ^{1}H-NMR($CDCl_3$):δ 7.16-7.66 (m, 5H, ArH), 8.33 (d,1H, J = 2Hz, H-1), 8.96 (m, 1H, H-10). MS, m/z(%): 286(M^+, 100), 288(34), 258(28), 223(47), 196(17), 179(13), 134(23), 108(28), 90(53).

2-Chloro-8-methyl-12H-benzothiazolo[2,3-b]quinazolin-12-one (4g): Irradiation time 19 hours, yield 66%, m.p. 208-210°C (lit[10]. m.p. 209-210°C). IR: 1690, 1580, 1570, 1550, 1470, 820 cm^{-1}. ^{1}H-NMR($CDCl_3$):δ 2.42 (s, H, CH_3), 7.23 (dd, J = 9.3Hz, J' = 2.0Hz, 1H, H-9), 7.37 (s, 1H, H-7), 7.55 (d, J = 8.8Hz, 1H, H-4), 7.66 (dd, J = 8.8 Hz, J' = 2.4Hz, 1H, H-3), 8.29 (d, J = 2.5 Hz, 1H, H-1), 8.76 (d, J = 8.3Hz, 1H, H-10).

Irradiation of disulphide 5a: The disulphide **5a** (575 mg, 1mmol) was dissolved in chloroform (40 ml) and methanol (130 ml) and irradiated at 300 nm for 32 hours. The chromatographic purification afforded the

product **4a**. Yield (240 mg) 48%; identified by m.p. 190-192°C, m.m.p. (with the previously obtained sample from compound **1a**) 190-192°C and superimposable IR. The disulphide **5b**, on irradiation for 14 hours likewise afforded **4c** (66%) identified by m.p, m.m.p. and superimposable IR.

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