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**Studies of Azo Derivatives of 8-Amino-10, 11-Dihydro-5H-Dibenzo (b,e) (1,4)-Diazepin-11-Thione : Part IV**

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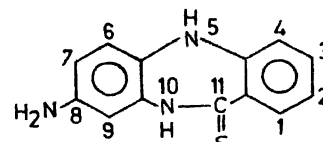
Manuscript received 16 October 1979, revised 13 October 1980, accepted 28 January 1981

**DIAZONIUM** salt of 8-amino-10, 11-dihydro-5H-dibenzo (b,e) (1,4)-diazepin-11-thione(I) was condensed with phenol, resorcinol,  $\alpha$ - and  $\beta$ -naphthols, 1-hydroxy-2-naphthoic and 3-hydroxy-2-naphthoic acids, separately and azo dyes (II~VII) were obtained.

In dibenzodiazepines, known as potent CNS drugs being used as antihistaminic, spasmolytic, antiparkinson and neuroplegic drugs<sup>1</sup> (Tarpan, Noveril and Clozapine<sup>2</sup> are some marketed drugs), aryl-azo group has been introduced. Azo dyes are also important class of pharmacological drugs<sup>2,3</sup>. Diazonium salt of I was condensed at 0-5° with phenol, resorcinol,  $\alpha$ - and  $\beta$ -naphthols, 1-hydroxy-2-naphthoic or 3-hydroxy-2-naphthoic acids separately and the corresponding azo dyes (II~VII) with yellow to red shades were obtained.

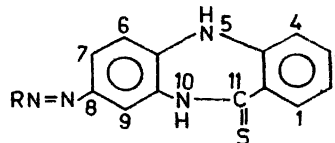
### Experimental

All the m.p.'s are uncorrected. Ir spectra were recorded on a Perkin-Elmer Infracord using KBr pellets. The purity of the azo dyes was ascertained by tlc using silica gel G as stationary phase and  $C_6H_6$  :  $C_2H_5OH$  :  $NH_3$  (7 : 2 : 1, solvent layer) as mobile phase.

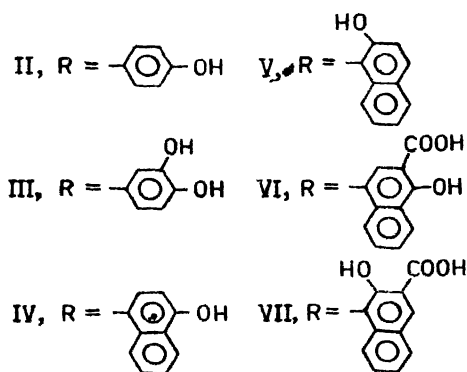


I

↓



II~VII



Chart—I

### Synthesis of Azo Dyes :

#### 8-p-Hydroxyphenylazo-10,11-Dihydro-11-Thione-5H-Dibenzo (b, e)-1,4-Diazepine-II-Thione :

To the diazotised solution of I\* (1.2050 g, 0.05M in 3 ml of conc. HCl, 2.0 ml of acetic acid and 0.8 g of sodium nitrite at 0-5°) alkaline solution of phenol (0.675 g in 5 ml of 10% NaOH) was gradually added with stirring keeping temperature range, 0-5°. The precipitated azo dye was filtered, washed with water and crystallised from ethanol (II, m.p. 285°, yield 0.9812 g, 56.72%,  $R_f$  0-59).

On similar lines azo dyes (III~VII) were obtained by condensing diazonium salt of I with

TABLE 1

Compound Number	Molecular Formula	Colour	m.p.	$R_f$	Yield	Visible Range		ir (cm) <sup>-1</sup>
						$\lambda_{max}$	$E_{max}$	
II	$C_{19}H_{14}ON_4S$	Red	285	0.59	56.72	400	11040	3120, 1610, 1575, 1390
III	$C_{19}H_{14}O_2N_4S$	Yellowish Brown	180	0.70	57.52	412	13350	3480, 1620, 1570, 1395
IV	$C_{23}H_{16}ON_4S$	Red	130	0.58	58.66	410	11400	3400, 1610, 1550, 1390
V	$C_{23}H_{16}ON_4S$	Red	230	0.56	59.83	428	13020	3440, 1600, 1500, 1375
VI	$C_{24}H_{16}O_2N_4S$	Reddish Yellow	250	0.62	55.29	460	11250	3400, 1680, 1570, 1390
VII	$C_{24}H_{16}O_2N_4S$	Yellow	200	0.65	60.92	457	13500	3400, 1640, 1580, 1405

\* For correspondence.

resorcinol,  $\alpha$ -naphthol,  $\beta$ -naphthol, 1-hydroxy-2-naphthoic acid and 3-hydroxy-2-naphthoic acid, separately.

The experimental values of nitrogen and sulphur percentage (in II~VII) are in general agreement with the theoretical values (0.3-0.5%). Melting points, yields, visible spectral data,  $R_f$  and colour of these azo dyes are compiled in Table 1.

#### Acknowledgement

The authors are thankful to Head, Chemistry Department, University of Rajasthan, Jaipur for providing necessary facilities and one of the authors (S. S.) thanks I.C.M.R. authorities for financial assistance.

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#### Search for New Antiviral Agents. Part-IV :

#### Synthesis of 2-morpholino/diethylamino-dithiocarbamyl-methyl-benzoxazinones and corresponding 3-substituted quinazolin-4-ones

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Manuscript received 12 April 1979, revised 5 December 1980,  
accepted 28 January 1981

A wide variety of sulphur containing compounds such as thiosemicarbazones<sup>1</sup>, thioureas<sup>2</sup>, phenothiazines<sup>3</sup>, thioamides<sup>4</sup>, thiazolinones<sup>5</sup> and thiazoles<sup>6</sup> have been proved to possess significant antiviral activity in the experimental animals. Further, Yoshioka *et al*<sup>7</sup> have recently established the antiviral activity of some dithiocarbamates. These observations prompted the authors to unite the quinazolinone and dithiocarbamyl moieties with a view to prepare compounds with modified antiviral activity, not studied so far.

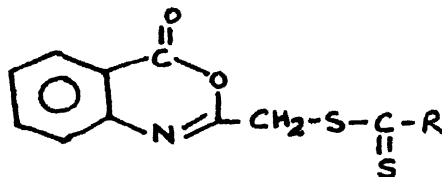
#### Experimental

##### 1. Morpholino/diethylamino dithiocarbamyl acetic acids :

These have been prepared following the method of Tiwari and Pandey<sup>8</sup>.

2.2-Morpholino/diethylamino-dithiocarbamyl-methyl-3,1 (4H)benzoxazine-4-ones : The procedure of Sammour *et al*<sup>9</sup> was followed. The compounds, thus synthesised, are listed in Table 1.

TABLE 1—2-MORPHOLINO/DIETHYLAMINO-DITHIOCARBAMYL-METHYL-BENZOXAZINE-4-ONES



Sl. No.	R.	m.p.	Mol. formula	Analysis Calcd.	Nitrogen found
1.	Morpholino	105°	C <sub>14</sub> H <sub>14</sub> N <sub>2</sub> O <sub>3</sub> S <sub>2</sub>	8.69%	8.41%
2.	Diethylamino	99-100°	C <sub>14</sub> H <sub>16</sub> N <sub>2</sub> O <sub>3</sub> S <sub>2</sub>	9.09%	9.26%

Note : All the melting points are uncorrected. Solvent used for recrystallization was ethanol.

3.2 - Morpholino/diethylamino - dithiocarbamyl - methyl-3-(aryl-substituted), 3,1 (4H) quinazolin-4-ones : A mixture of 2-morpholino/diethylamino-dithiocarbamyl-methyl-3, 1 (4H)-benzoxazine-4-one (0.01 mole) and substituted amine (0.01 mole) in 30 ml of dry pyridine was refluxed for 6 hours under anhydrous conditions. The reaction mixture was then poured in the ice cooled water containing dil HCl. The solid product that separated out, was washed repeatedly with water and recrystallized from dil ethanol. The various compounds thus, synthesised and listed in Table 2.

#### Antiviral Activity :

Materials and Methods : Four compounds (compound Nos. 1, 4, 6 and 9 of Table 2) were screened for their antiviral activity against Semliki forest virus (S.F.V.) on swiss albino mice weighing 20 gm (average weight). Each test compound (100 mg/kg body weight) was added to carboxy methyl cellulose (C.M.C.) solution and injected in test mouse through intraperitoneal route in two divided doses at 24 and 48 hrs after virus infection. Ten mice were used for each test compound and ten were kept as control. The mice were then observed for 15 days for the appearance of symptoms (paralysis) and death. Protection rate (survival percentage) and