

CHAPTER - 9

EXPERIMENTAL WORK.

E X P E R I M E N T A L W O R K

CHEMICALS

- 1) 4,4' - Diaminostilbene 2,2' disulphonic

acid :

This compound was purified by recrystallization from acetone/alcohol and its purity ^{was} checked by spectral methods and M.P. 228⁰c

- 2) Cyanuric chloride :

Cyanuric chloride was used of West-German Degausse ^{was}
^
company.

- 3) Aniline :

It was used of B . D . H . (A . R .) grade

- 4) ~~o~~-Toluidine :

It was used of B . D . H . (A . R .) grade

- 5) ~~p~~-Toluidine :

It was used of B . D . H . (A . R .) grade

- 6) Chlorosulphonic acid :

B . D . H . (A . R .) grade was used

- 7) ~~o~~-Chloroaniline :

B . D . H . (A . R .) grade was used

- 8) ~~o~~ naphthylamine :

B . D . H . (A . R .) grade was used

9) Diethanol amine

It was used of B . D . H . (A . R .) grade

Instruments used for spectral study

- 1) U.V. - Double beam spectrophotometer Hitachi, -UV-330 spectrophotometer
- 2) I.R. Spectra were recorded in KBr pellets on a "Perkin-Elmer-783 infrared spectrophotometre".
- 3) Fluorescence Shimadu-Double monochromator spectrofluorophotometer RF-540

SCHEME I : SYNTHESIS OF 4,4¹ DIAMINOSTILBENE

2,2¹ DISULPHONIC ACID :

The synthesis of 4,4'-diaminostilbene 2,2'-disulphonic acid has been reported⁹¹. This was prepared by three-step synthesis from p-nitrotoluene. The latter was first sulphonated with 26% oleum at 55-60⁰c to give 4-nitrotoluene-2-sulphonic acid.

A paste of 4-nitrotoluene-2-sulphonic acid was dissolved in a large amount of water and neutralised with sodium hydroxide solution. The sodium salt of 4-nitrotoluene-2-sulphonic acid was then oxidized with a solution of sodium hypochlorite at 75-80⁰c under simultaneous addition of sodium hydroxide solution.

After termination of the oxidation process the reaction mixture was neutralised with concentrated hydrochloric acid, cooled and salted out. The separated product was isolated by filtration on a filterpress. The paste of

4,4'-dinitrostilbene- 2,2' - disulphonic acid was then reduced by adding it gradually into an aqueous suspension of iron fillings etched with hydrochloric acid, at a temperature of about 100°C.

The reaction mixture was made alkaline with sodium hydroxide and freed of iron-containing sludge on a filter press. From the filtrate 4,4'-diamino stilbene - 2,2' disulphonic acid was precipitated by acidification with sulphuric acid. The product was filtered off, for further work product was usually used directly in the form of paste.

Preparation of sodium salt of 4,4' - diaminostilbene - 2,2' disulphonic acid :

4,4' Diaminostilbene 2,2' disulphonic acid (11 gms) was added in 25 ml distilled water in 250 ml beaker. The mixture was well stirred and equimolar quantity of sodium carbonate (17.2 gms) was added. The mixture was stirred and heated till clear solution ^{was} obtained.

Yield - 80% M.P. -> Decomposes at 226-228°C

Analytical data :

	C	H	N
Calculated	40.30	3.60	7.22
Found	40.30	3.58	7.21

Spectral data :

1> U.V. - A=210 nm, B=335 nm

2> I.R. - NH₂-3450 - 3250 cm⁻¹

C=C -1600-1500 cm⁻¹

-C-N 1200-1020 cm⁻¹

-C-H Stretching 3030 - 3080 cm⁻¹

U V ABSORPTION SPECTRUM OF SODIUM SALT OF
4,4'-DIAMINO STILBENE 2,2'-DISULPHONIC ACID.

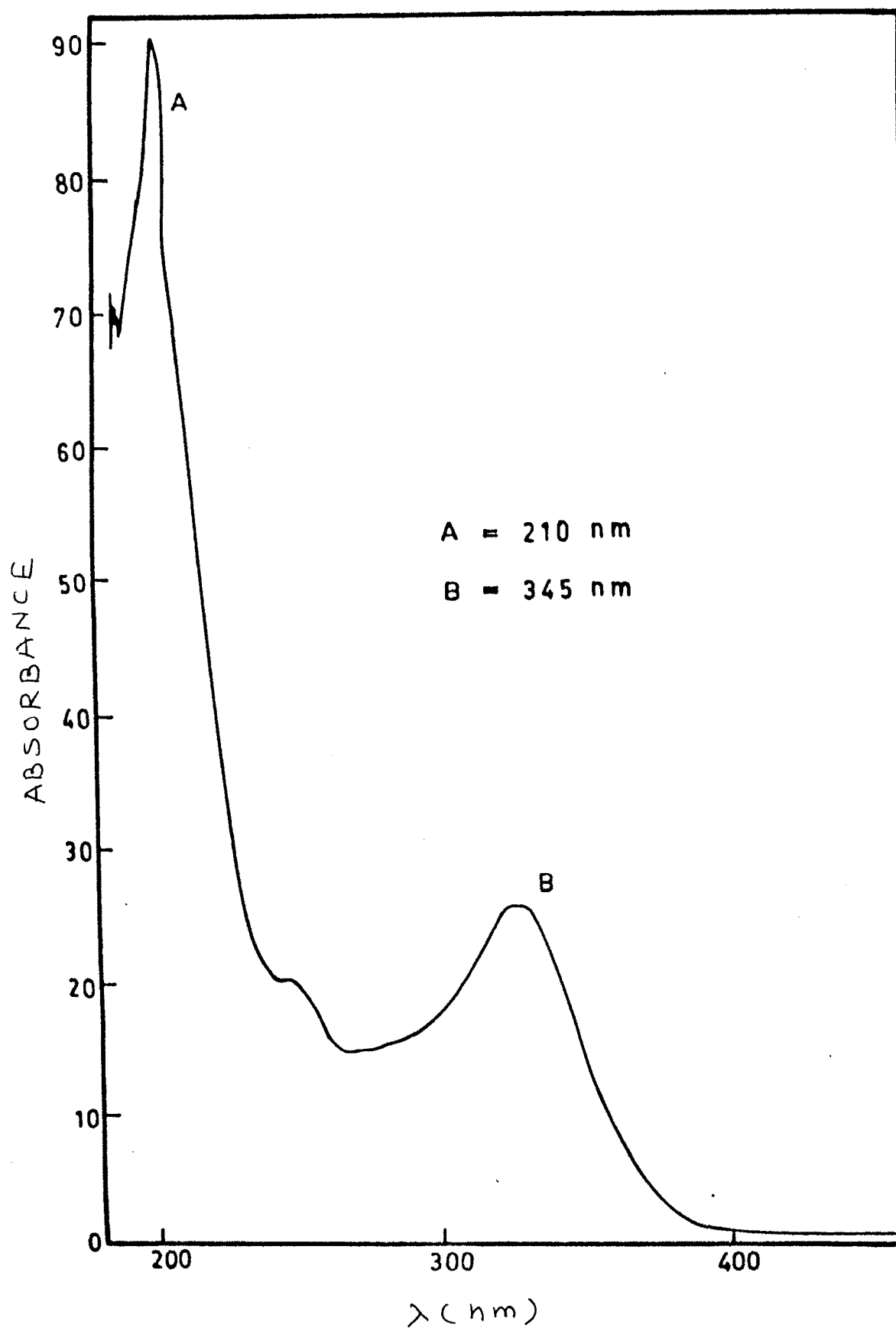


FIG.

I R SPECTRUM OF SODIUM SALT OF 4,4'-DIAMINOSTILBENE-2,2'-DISULPHONIC ACID .

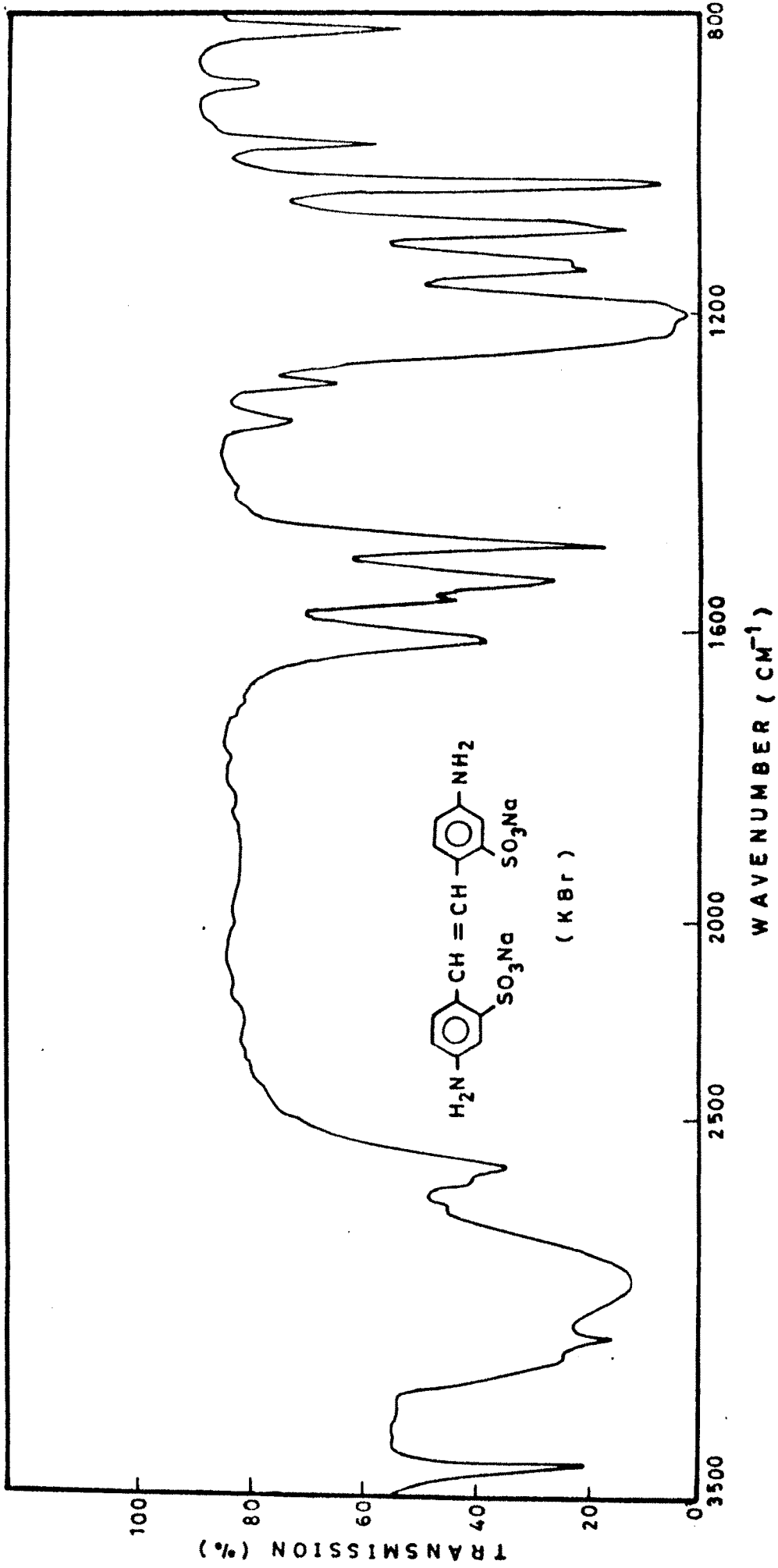


FIG.

Scheme II

1> Acetylation of Amines :

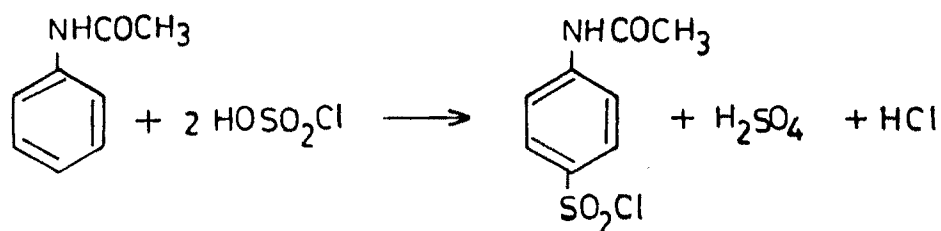
Acetanilide was prepared by refluxing a mixture of aniline (20 ml), acetic anhydride (20 ml) and glacial acetic acid (20 ml) gently for 30 minutes and then pouring the hot liquid in a thin stream into the cold water with continuous stirring. The crude product thus obtained was recrystallised from very dilute water alcohol mixture.

Yield was 18 gms and m.p. 114°C . The same procedure was used for the acetylation of *o*-toluidine and *p*-toluidine.

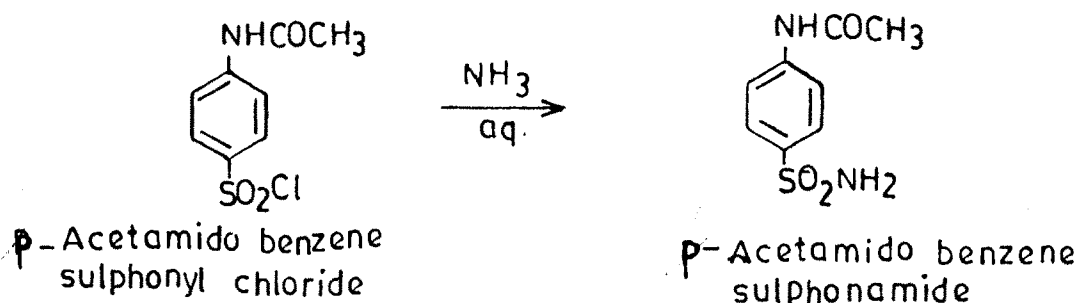
2> General Procedure for the Preparation of Substitued Sulphonamides.

Preparation of *p*-Amino benzene Sulphonamide. This compound was synthesised by using following three steps.

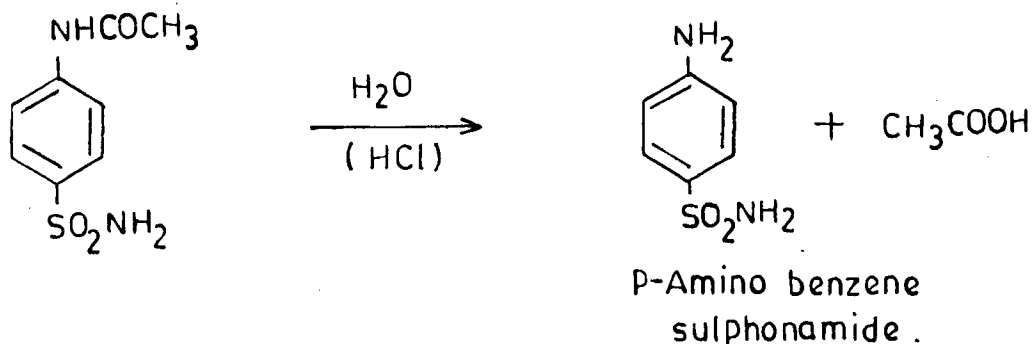
I) Treatment of acetanilide with excess of chlorosulphonic acid afforded *p*-acetamido benzene sulphonyl chloride. m.p. 149°



II) This was converted by aqueous ammonia into *p*-acetamidobenzene sulphonamide m.p. 218°



III> By boiling with dilute hydrochloric acid the protecting acetyl group was removed without hydrolysing the sulphonamido group. The liberated sulphonamide was passed into solution as the hydrochloride and the free base (P-amino benzene sulphonamide) was obtained by neutralisation with sodium bicarbonate. m.p. 163°C



Procedure :

I> Preparation of P-Acetamidobenzene sulphonyl chloride.

20 gms of dry acetanilide were placed in the 500 ml bolt head flask with two holded cork carrying a dropping funnel and a reflux condenser. 50 ml (90 gms) of a good grade chlorosulphonic acid was added in small portions and the flask was shaken from time to time to ensure thorough mixing. When the additon has been made, the reaction mixture was heated on a water bath for 1 hour in order to complete the reaction. After cooling the oily mixture was poured in a thin stream with stirring into 300 gms of crushed ice contained in a 1 litre beaker. The reaction mixture was stirred for several minutes in order to obtain an even suspension of the granular white solid. The P-acetamido benzene sulphonyl chloride was filtered at the pump and washed with a little cold water and recrystallised from a mixture of equal volumes of benzene and acetone m.p. 149°C

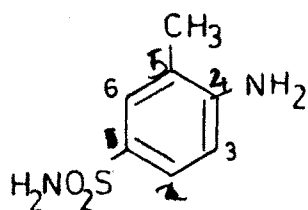
II> Preparation of P-Acetamido benzene sulphonamide

The above P-acetamidobenzene sulphonyl chloride was taken in the reaction flask and a mixture of 70 ml of concentrated ammonia solution (sp. gr. 0.88) and 70 ml of water was added. The reaction mixture was heated with occasional swirling to just below the boiling point for about 15 minutes. The reaction mixture was cooled and just acidified with dilute sulphuric acid. The product was collected on a buchner funnel, washed with little cold water and drained as completely as possible m.p. 218⁰C.

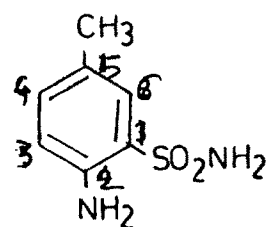
III) Preparation of P-Amino benzene sulphonamide.

The crude P-acetamido benzene sulphonamide was transferred to a 500 ml flask and a mixture of 10 ml of concentrated hydrochloric acid and 30 ml of water was added. The mixture was boiled gently under reflux for 40 minutes. To the cooled solution 2 gms of decolouring carbon was added and heated the mixture to boiling and filtered with suction through a hardened filter paper. The filtrate was placed in a litre beaker and cautiously added 16 gms of solid sodium bicarbonate in portions with stirring. (sodium bicarbonate was added until the reaction mixture became neutral) sulphanilamide was filtered & recrystallised from water or from alcohol m.p. 163⁰C.

The same procedure was used for the synthesis of sulphanomides from ortho and para acetyl toluidines.



o-Toluidine Sulphonamide
5 methyl 4 amino benzene
sulphonamide



p-Toluidine Sulphonamide
5 methyl 2 amino
sulphonamide

IR SPECTRUM OF P-AMINO BENZENE SULPHONAMIDE .

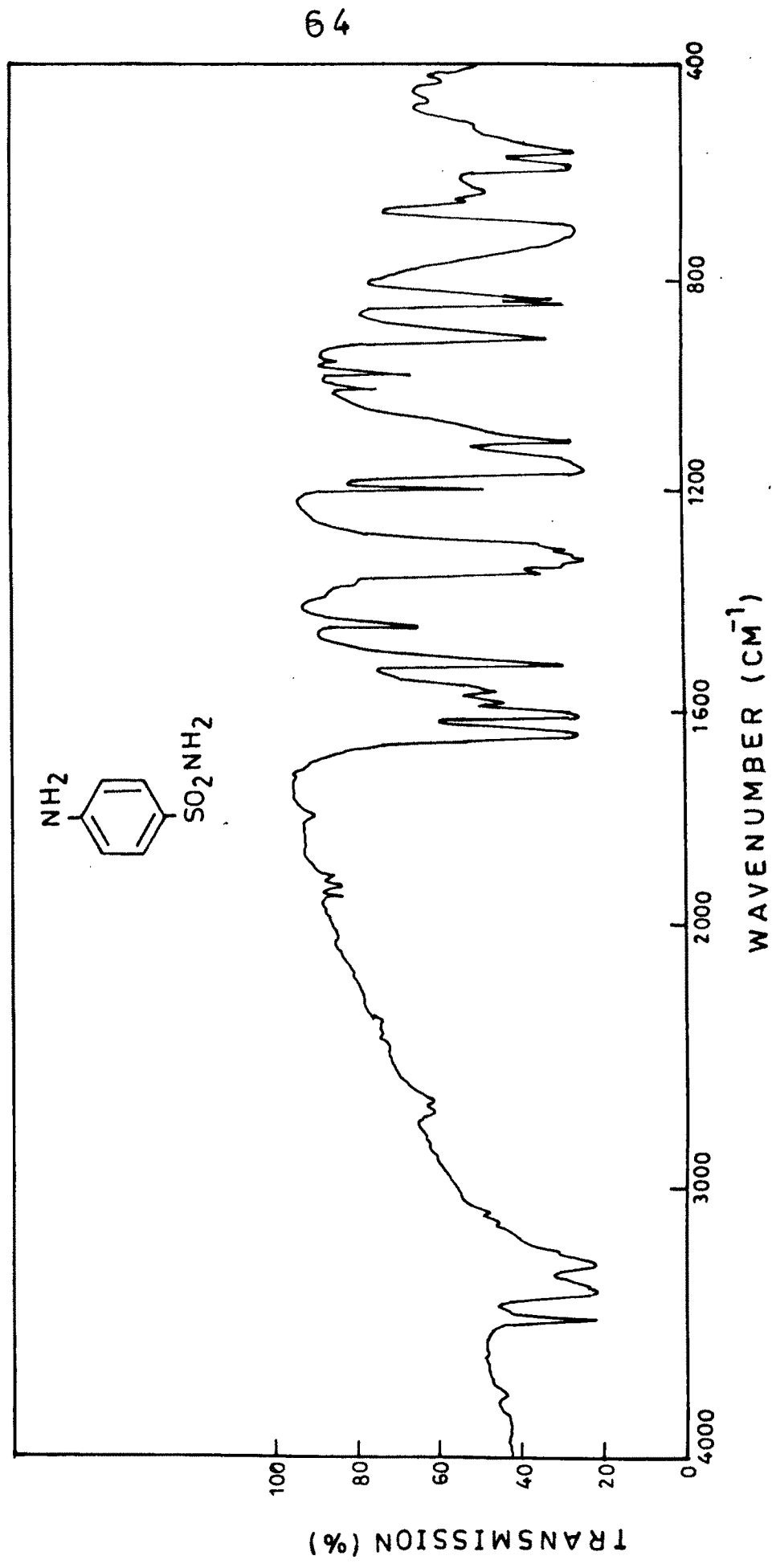
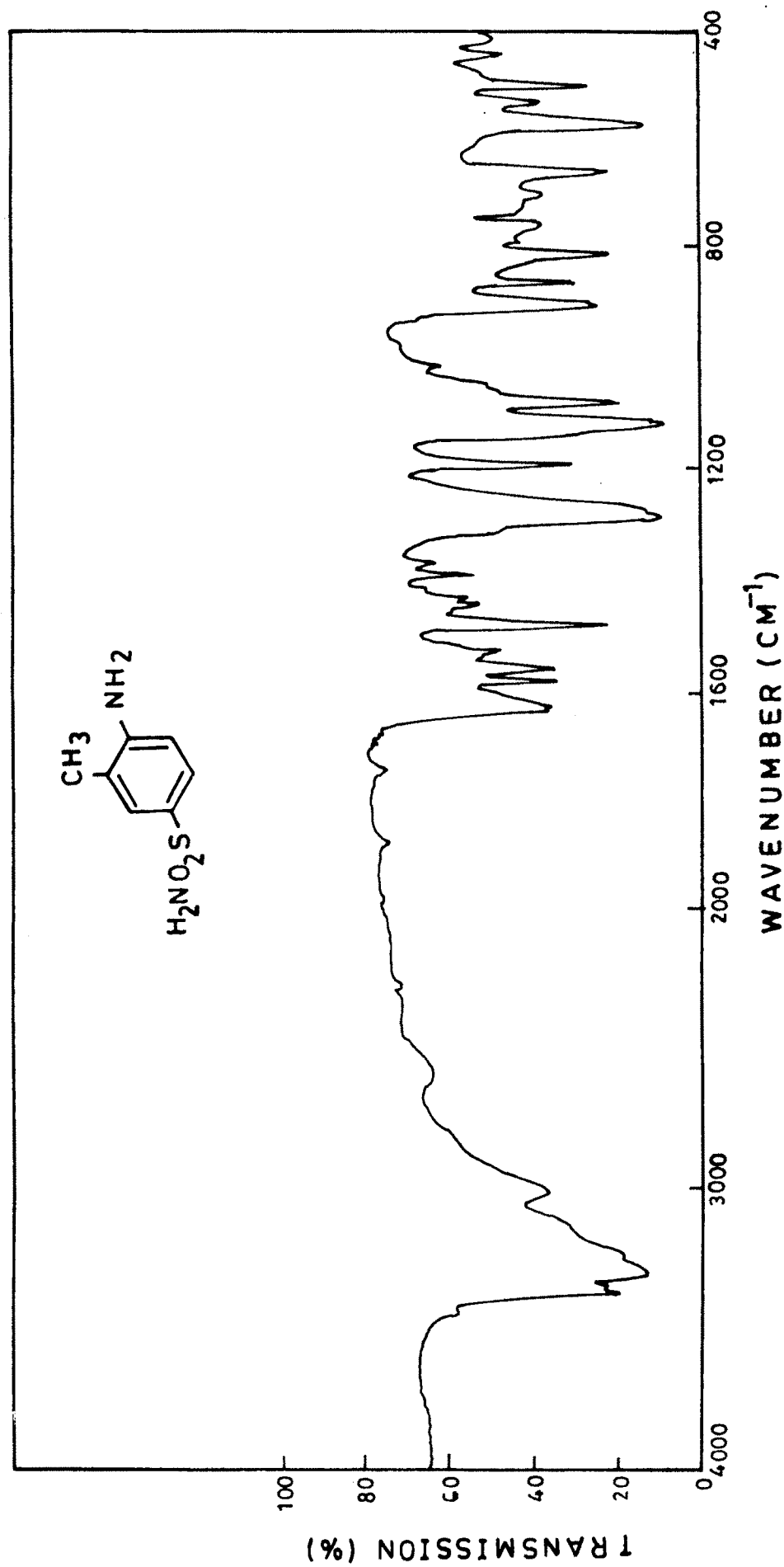
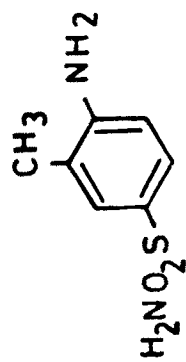


FIG. NO.

IR SPECTRUM OF o-TOLUIDINE SULPHONAMIDE OR
2-AMINO-1-METHYL-5-SULPHONAMIDO BENZENE .



TRANSMISSION (%)

WAVENUMBER (CM⁻¹)

FIG. NO.

IR SPECTRUM OF P-TOLUIDINE SULPHONAMIDE OR
4-AMINO-1-METHYL-3-SULPHONAMIDO BENZENE .

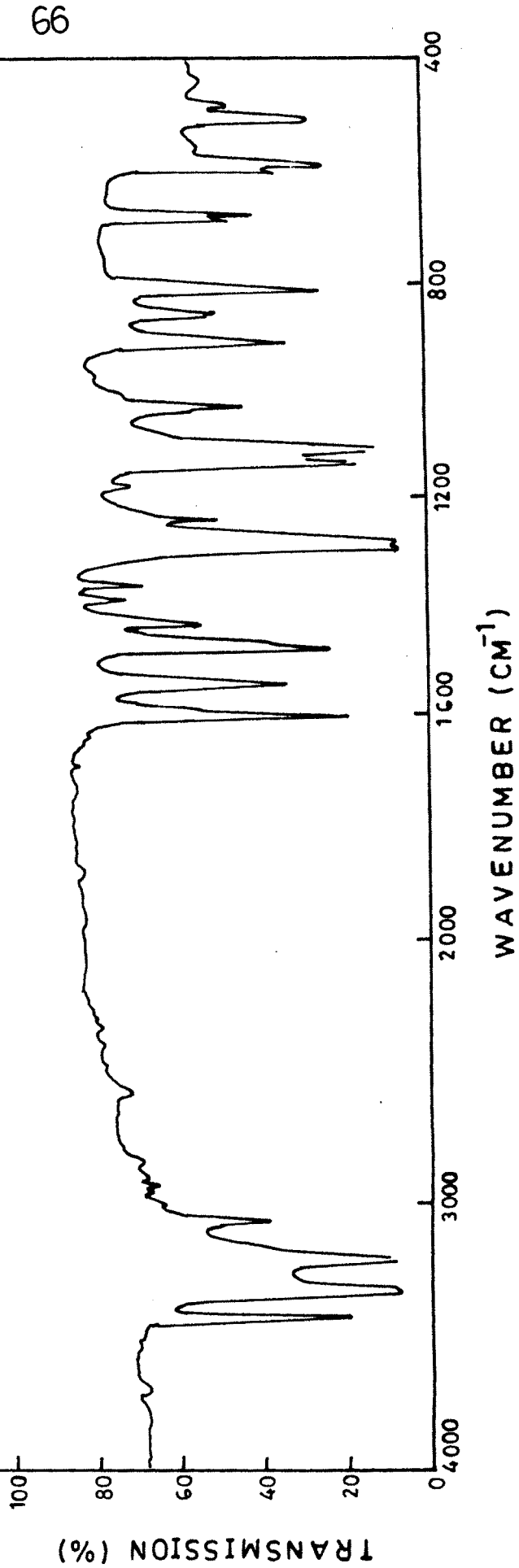
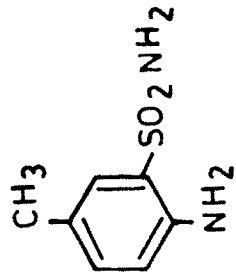
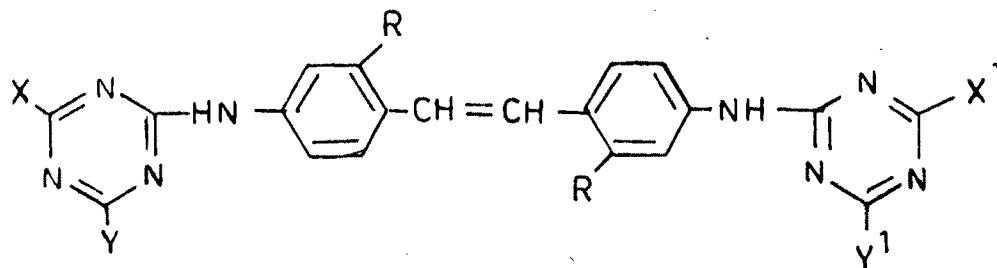


FIG.NO.

FLUORESCENT BRIGHTENING AGENTSGeneral structure :

$X = X'$, $Y = Y'$, $R = SO_3Na$

Symmetrical fluorescent brightening agent,

where $X = X'$, $Y = Y'$ 92

This type of fluorescent brightener was storage stable at -30 to $+50^{\circ}C$ and suitable for use on cellulosic fibres or in the paper making.

SYNTHESIS OF FLUORESCENT BRIGHTENER NO 1

STEP 1 - SYNTHESIS OF 6- (P-AMINO BENZENE SULPHONAMIDO) -2,4 DICHLORO 1-3-5 TRIAZINE.

P-Amino benzene sulphonamide (8.6 gms, 2 mole) was dissolved in 0.01 M sodium hydroxide solution (PH = 10.8). It was cooled in icebath at $0-5^{\circ}C$. Beaker was equipped with mechanical stirrer. Cyanuric chloride (10 gms, 2 mole) was dissolved in aqueous acetone (1:3), 25 ml and then added in small quantity at a time and reaction was stirred vigorously. Reaction was continued after adding all quantity of cyanuric chloride. The PH of soln quickly fell

from 10.8 to 2.5 . The reaction mixture was stirred for one hour.

STEP 2 - SYNTHESIS OF 4,4 BIS [6 -(P-AMINO BENZENE SULPHONAMIDO) - 4 - CHLORO -1,3,5 TRIAZIN - 2-YL] AMINOSTILBENE -2,2 DISULPHONIC ACID.

A paste of sodium salt of 4,4' diamino stilbene - 2,2' - disulphonic acid (8 gms 1 mole) was added to the above reaction product at room temperature. Addition was made slowly and dropwise with continuous stirring. The completion of reaction [condensation of primary amine with cyanuric chloride] was checked by testing unreacted aromatic primary amine by diazotization method.

STEP 3 - SYNTHESIS OF 4,4' BIS[6-(P-AMINO BENZENE SULPHONAMIDO) -4- DIETHANOLAMINO -1,3,5 TRIAZIN -2-YL] AMINOSTILBENE 2,2' DISULPHONIC ACID.

Reaction mixture was heated to 85-90⁰C. Diethanolamine (6.5 gms. 2 mole) was added dropwise. Stirring was continued for 30-40 minutes. (PH-9) Reaction mixture was cooled. It was salted out with sodium chloride and filtered, washed with acetone, dried and the crude product was weighed. Fine yellow coloured granular powder was obtained.

Yield - 18 gms

M. P. - Decomposes at 290⁰C.

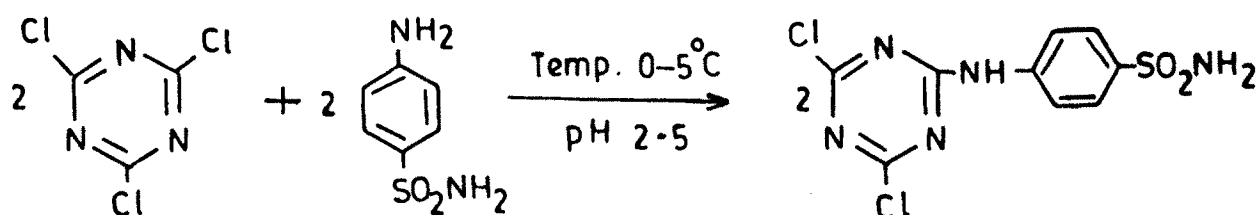
Analytical data	percentage of elements		
	C	H	N
Found	42.90	3.91	17.50
Calculated	42.93	3.93	17.53

Spectral Analysis

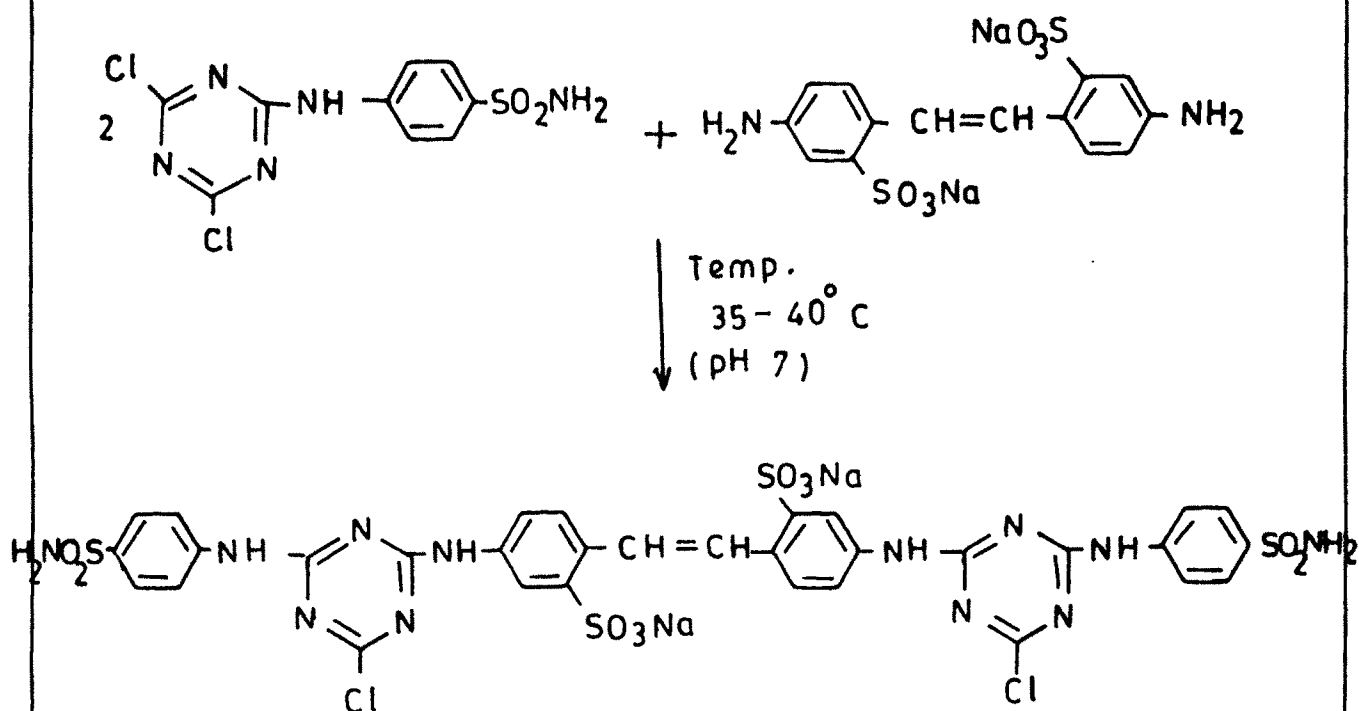
- 1) U.V. - A= 280 nm . B= 350 nm
- 2) IR - NH (stretching) 3400 cm^{-1}
- C= N (stretching) $1500 - 1600 \text{ cm}^{-1}$
- S= O (stretching) 1370 cm^{-1}
- S= O (stretching) 1170 cm^{-1}
- C= C (stretching) $1600-1650 \text{ cm}^{-1}$

REACTIONS OF FLUORESCENT BRIGHTNER NO.1

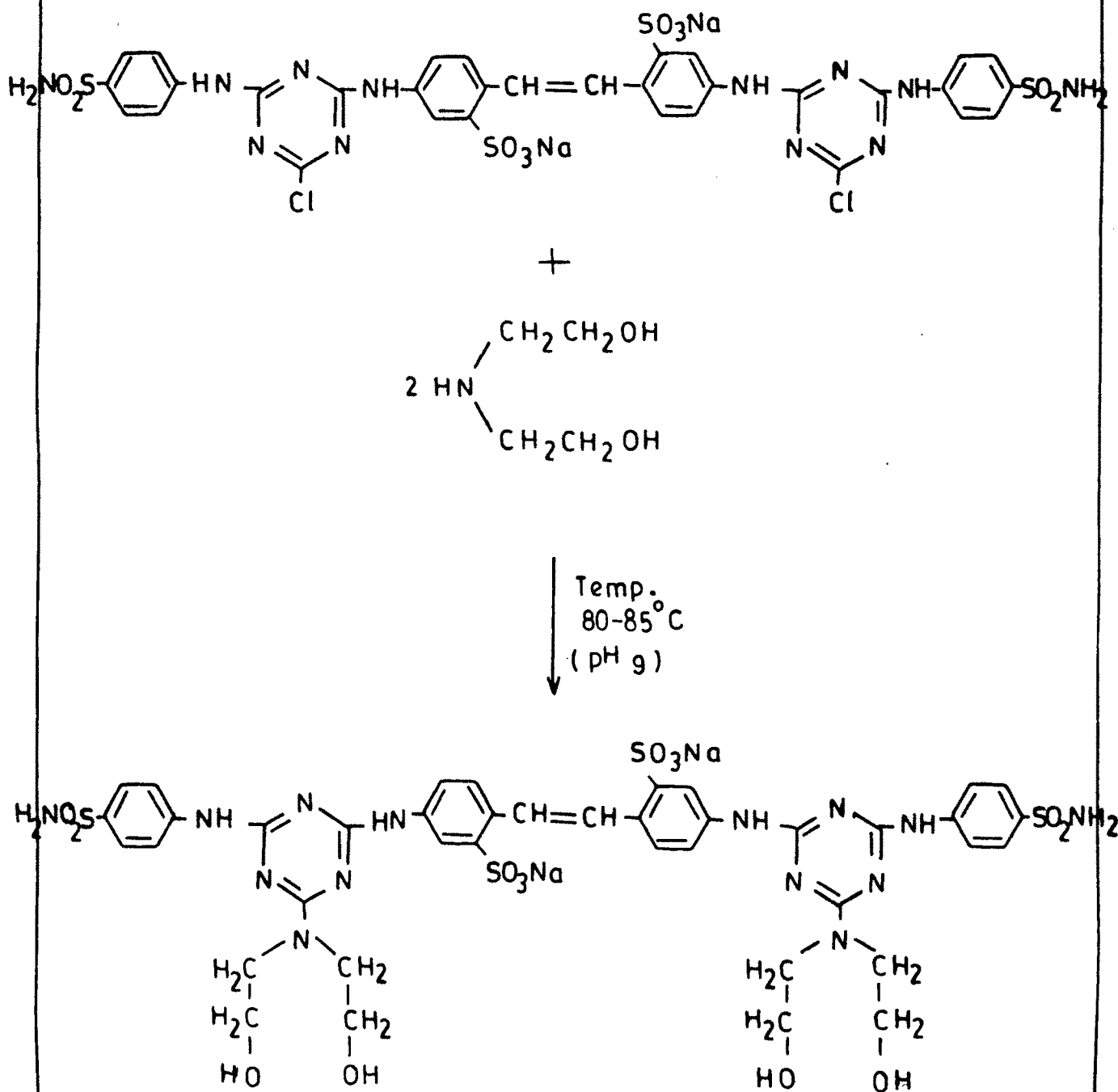
STEP I - SYNTHESIS OF 6 (P-AMINO BENZENE SULPHONAMIDO) 2,4 DICHLORO 1,3,5-TRIAZINE .



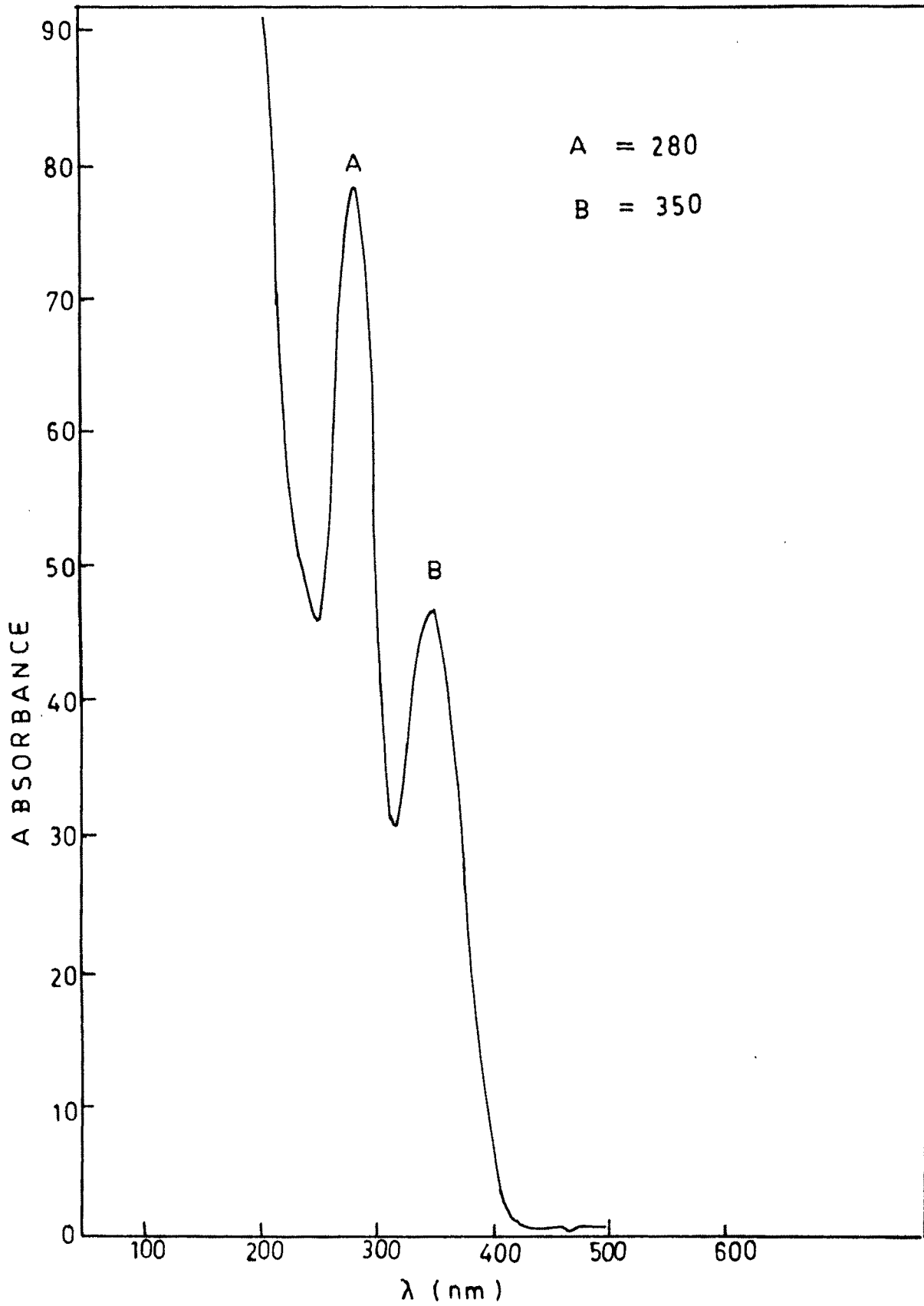
STEP II : SYNTHESIS OF 4,4' BIS [6 (P-AMINO BENZENE SULPHO - NAMIDO) -4- CHLORO-1,3,5 TRIAZIN-2-YL] AMINO STILBENE-2,2' DISULPHONIC ACID .



STEP III - SYNTHESIS OF 4,4' BIS [6 (P-AMINO BENZENE
SULPHONAMIDO) 4-DIETHANOL AMINO -1,3,5 -
TRIAZIN-2-YL] AMINO STILBENE 2,2'-DISULPHONIC ACID.



FLUORESCENT BRIGHTENER NO.1



UV ABSORPTION SPECTRUM OF F. B. A. NO. 1

IR SPECTRUM OF CONDENSATION PRODUCT OF P-AMINO BENZENE SULPHONAMIDE
AND CYANURIC CHLORIDE .

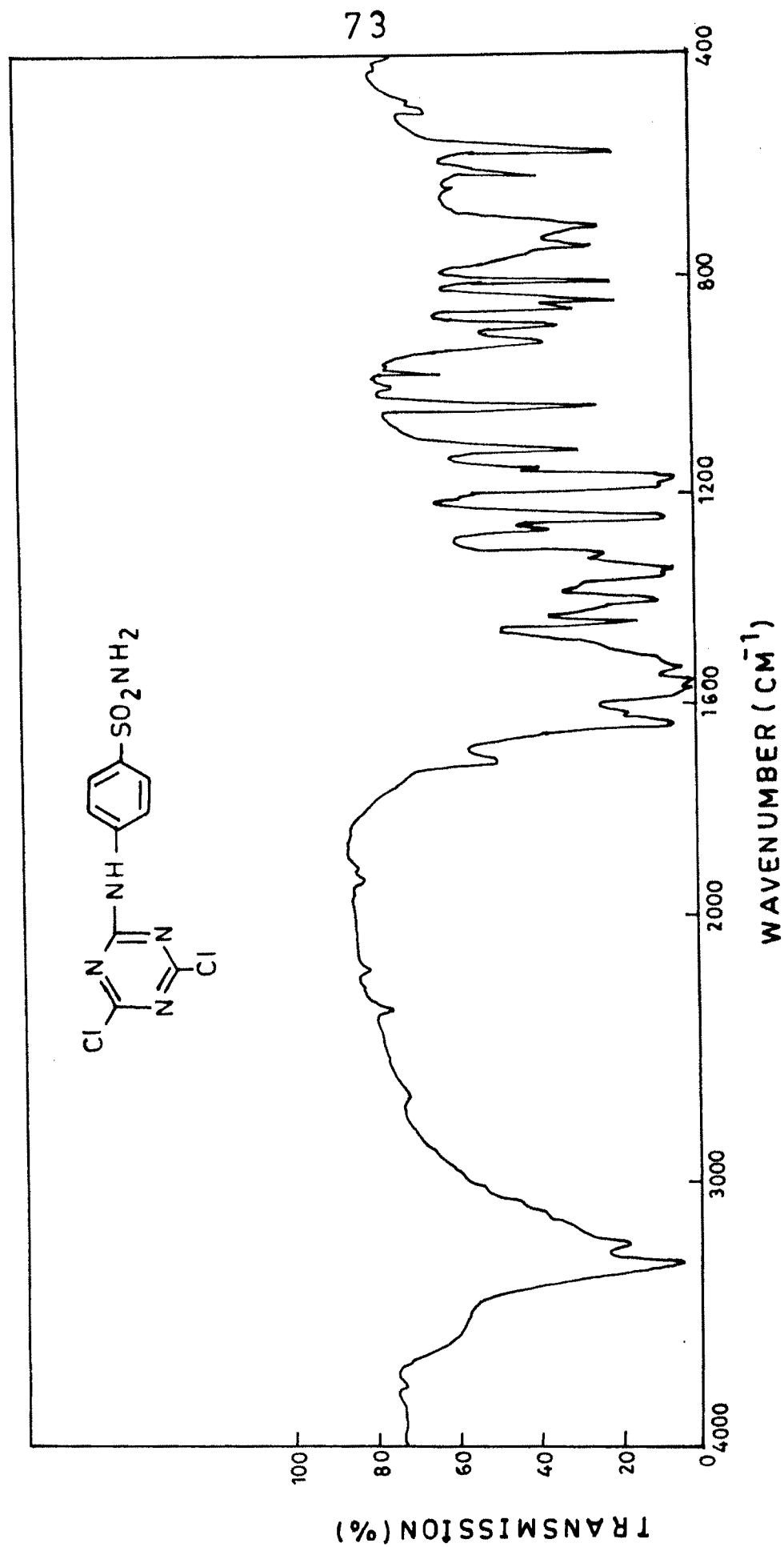


FIG. NO.

SYNTHESIS OF FLUORESCENT BRIGHTENER NO. 2

STEP 1 - SYNTHESIS OF 6 - (O-TOLUIDINE SULPHONAMIDO) 2,4
DICHLORO 1-3-5 TRIAZINE.

O-Toluidine sulphonamide (9.3 gms , 2 mole) was dissolved in 0.01 M sodium hydroxide solution (PH = 10.8). It was cooled in ice bath at 0-5°C. Beaker was equipped with mechanical stirrer. Cyanuric chloride (10 gms , 2 mole) was dissolved in aqueous acetone (1:3) 25 ml & then added in small quantity at a time and reaction was stirred vigorously. Reaction was continued after adding all quantity of cyanuric chloride. The PH of solution quickly fell from 10.8 to 2.5 . The reaction mixture was stirred for one hour.

STEP 2 - SYNTHESIS OF 4,4' BIS [6(O-TOLUIDINE
SULPHONAMIDO) . 4 CHLORO -1-3-5 TRIAZIN -2 -YL] AMINO
STILBENE 2,2' DISULPHONIC ACID.

A paste of 4,4' diaminostilbene 2,2' disulphonic acid (8 gms , 1 mole) was added in a above reaction mixture at room temperature. Addition was made slowly and dropwise. Condensation of DASDSA with cyanuric chloride required 2-3 hours. second chlorine atom was replaced at 35-40°C. To ensure reaction was completed [condensation of primary amine with cyanuric chloride] was checked by testing unreacted aromatic primary amine by diazotization method.

STEP 3 - SYNTHESIS OF 4,4' BIS [6 (O-TOLUIDINE
SULPHONAMIDO) -4- DIETHANOL AMINO 1,3,5 TRIAZIN -2-YL]

AMINO STILBENE -2,2' DISULPHONIC ACID.

Reaction mixture was heated to raise the temperature 85-90⁰C. As the third chlorine atom of cyanuric chloride was difficult to react, diethanol amine (6.5 gms 2 mole) was added in dropwise. stirring was continued for an hour. (PH =9). Reaction mixture was cooled. It was salted out with sodium chloride and filtered. dried with acetone. weighed the crude product.

Yield - 16 gms

M. P. - Decomposes on heating above 300⁰ -

Analytical data	percentage of element		
	C	H	N
Experimental	43.94	4.16	17.0
calculated	43.97	4.19	17.1

Spectral data :

1) U.V. A= 250 nm B= 350 nm

2) I.R. NH (stretching) -3400 cm⁻¹

C= N (stretching) 1500 - 1600 cm⁻¹

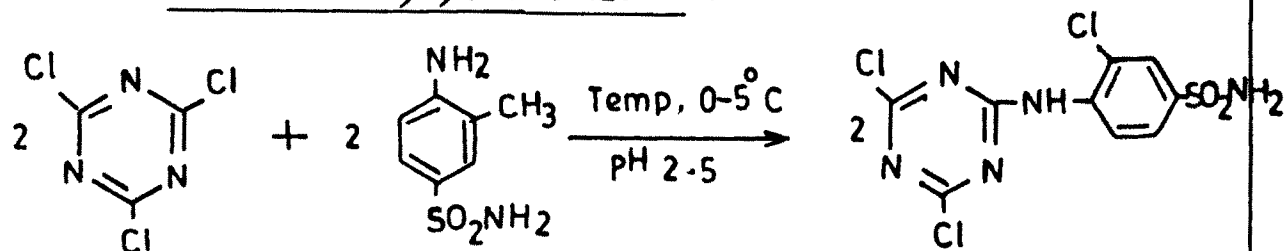
S= O (stretching) -1370 cm⁻¹

S= O (stretching) -1170 cm⁻¹

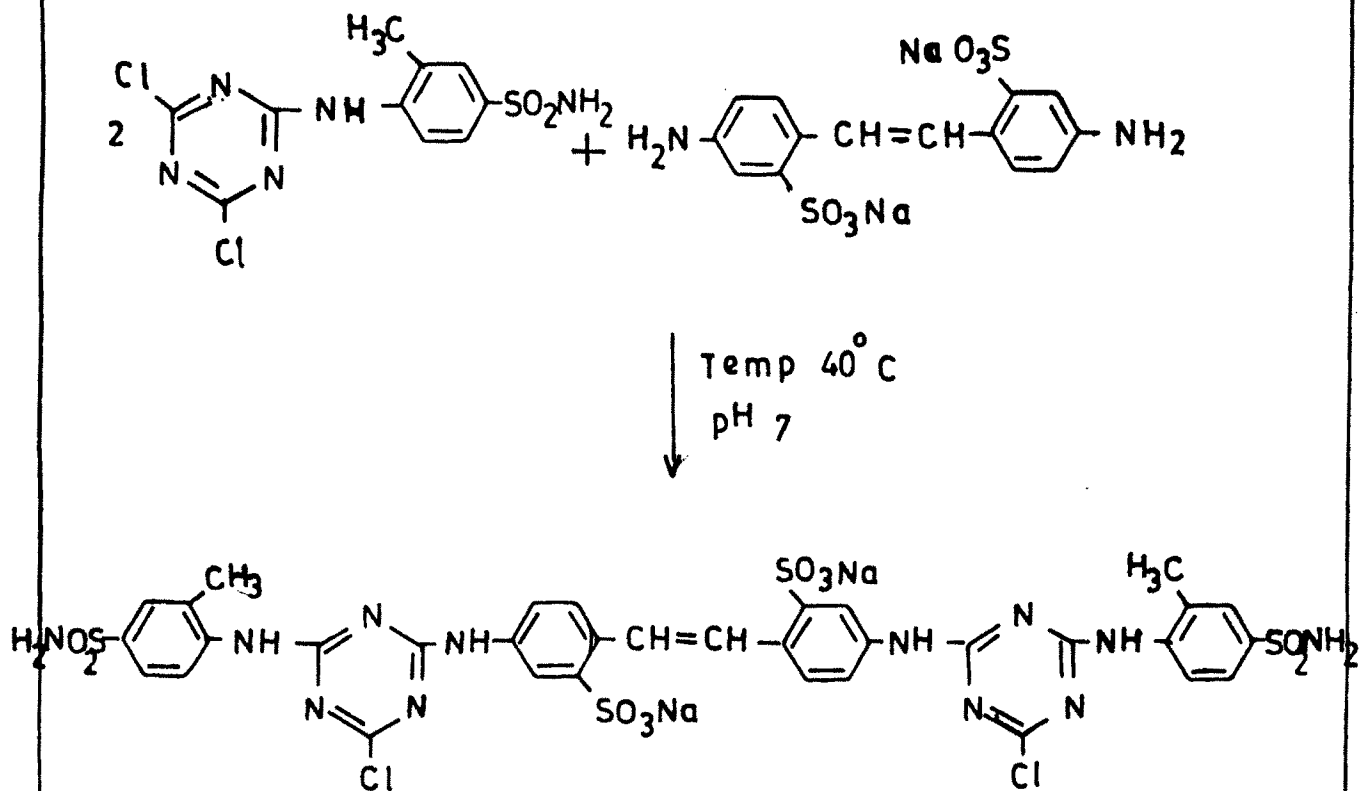
C= C (stretching) -1600 - 1650 cm⁻¹

REACTIONS OF FLUORESCENT BRIGHTENER NO. 2

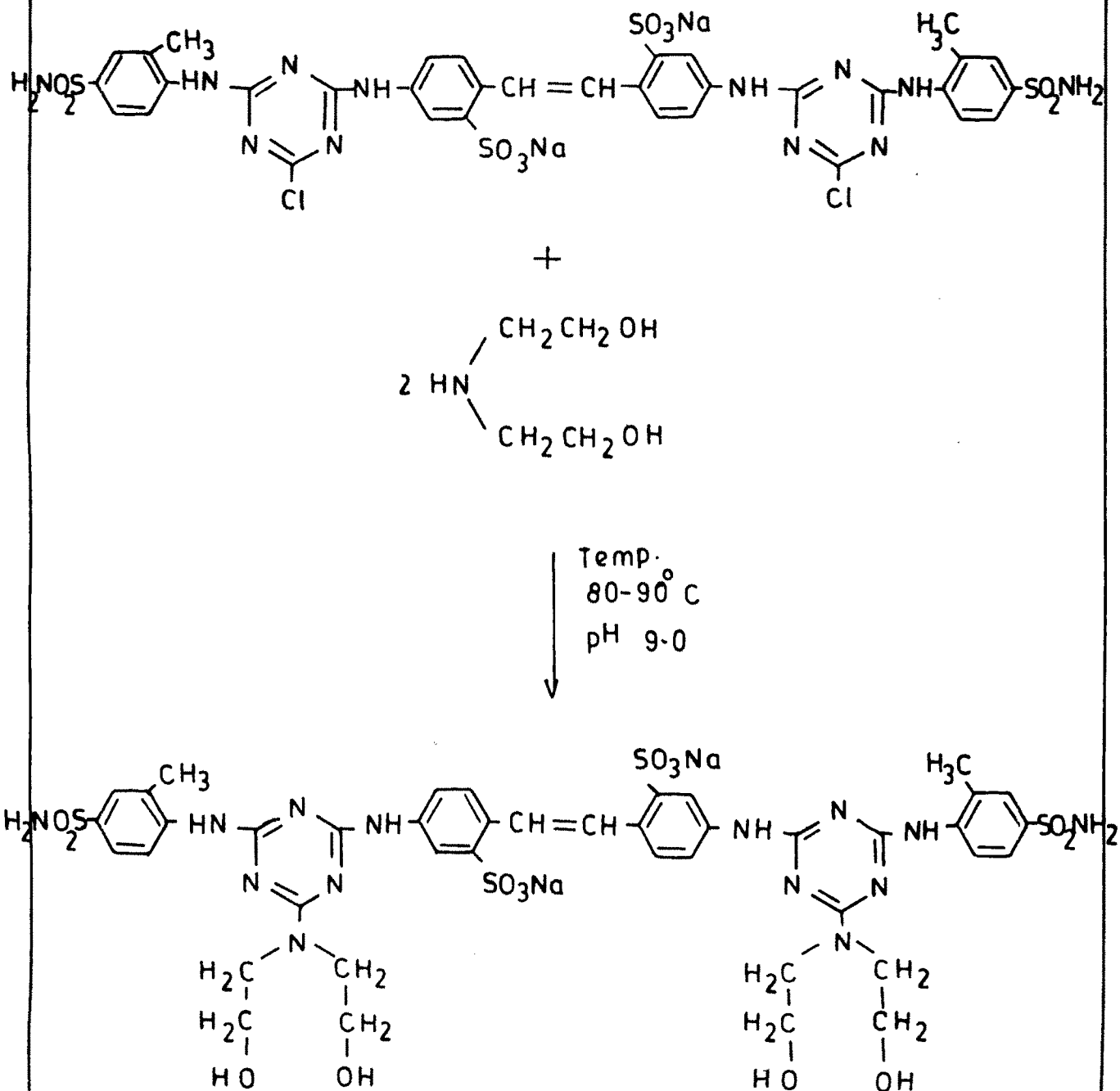
STEP I : SYNTHESIS OF 6 (o-TOLUIDINE SULPHONAMIDO) 2,4 -
DICHLORO-1,3,5 TRIAZINE



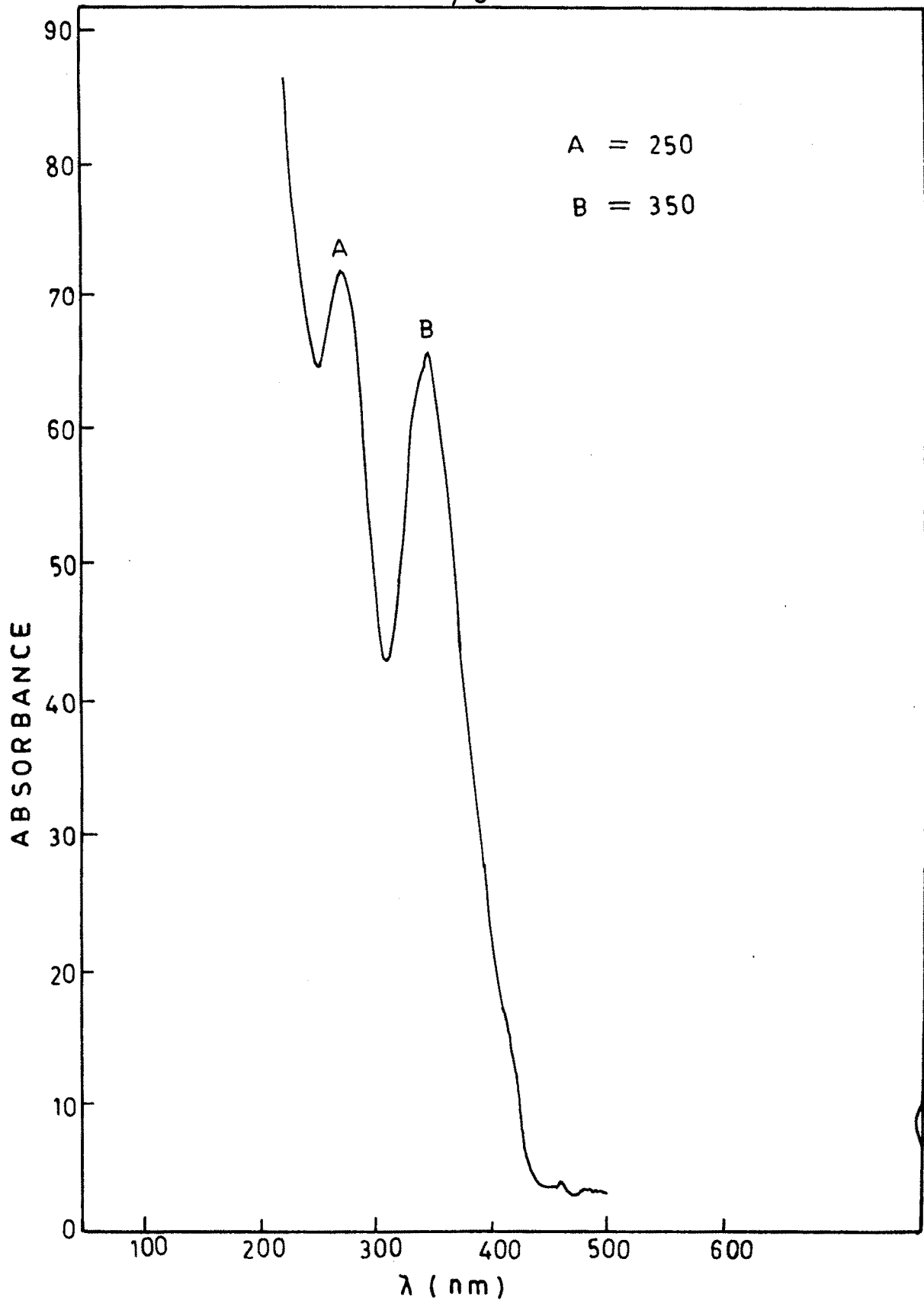
STEP II : SYNTHESIS OF 4,4' BIS [6 (o TOLUIDINE SULPHONAMIDO)
4-CHLORO-1,3,5-TRIAZIN-2-YL] AMINO STILBENE 2,2'
DISULPHONIC ACID .



STEP III - SYNTHESIS OF 4,4'-BIS [6 (o-TOLUIDINE SULPHONAMIDO) -MIDO] -4-DIETHANOLAMINO 1,3,5 TRIAZIN-2-YL] AMINOSTILBENE 2,2' DISULPHONIC ACID .



FLUORESCENT BRIGHTENER NO-2



UV ABSORPTION SPECTRUM OF F. B. A. NO. 2

I.R. SPECTRUM OF 6-O-TOLUIDINE SULPHONAMIDO - 2, 4 DICHLORO - 1, 3, 5-TRIAZINE .

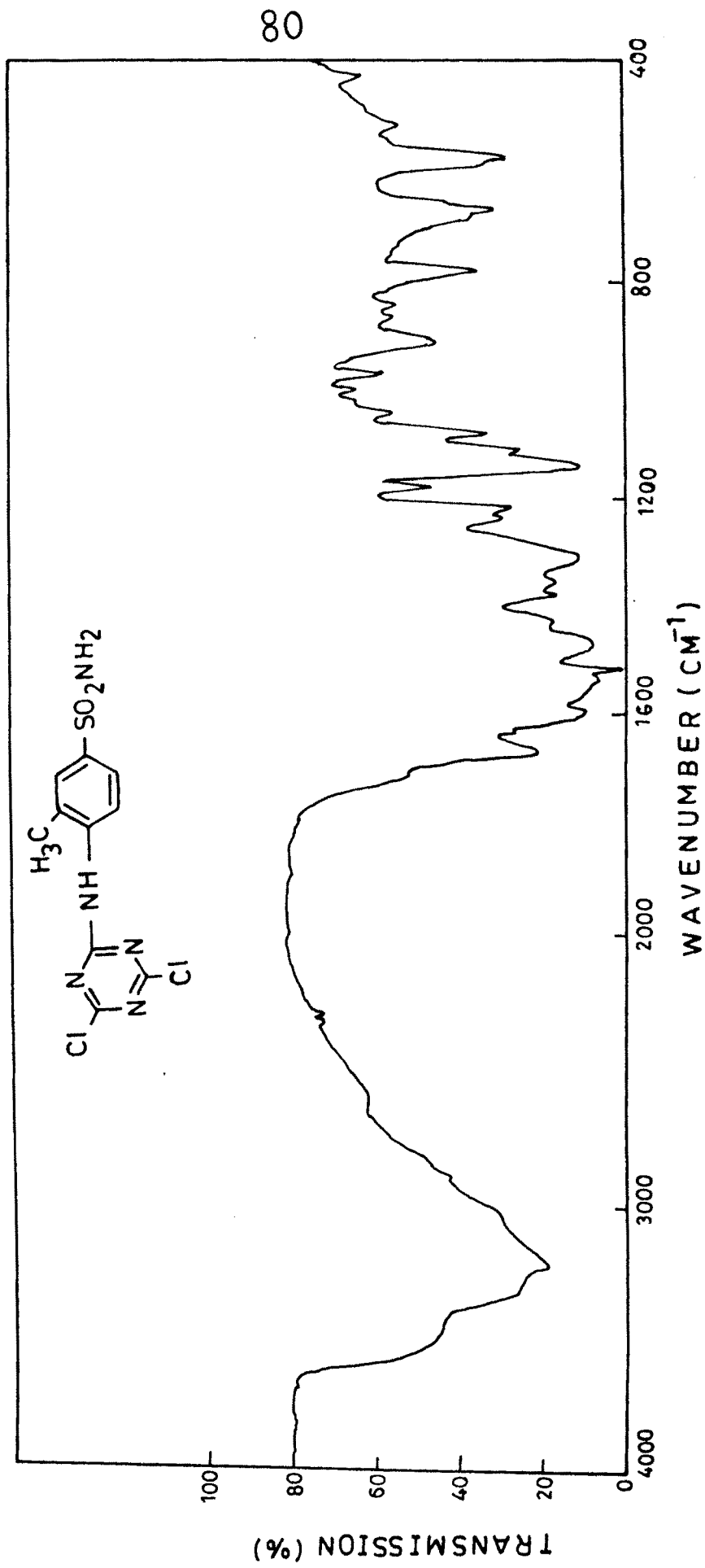
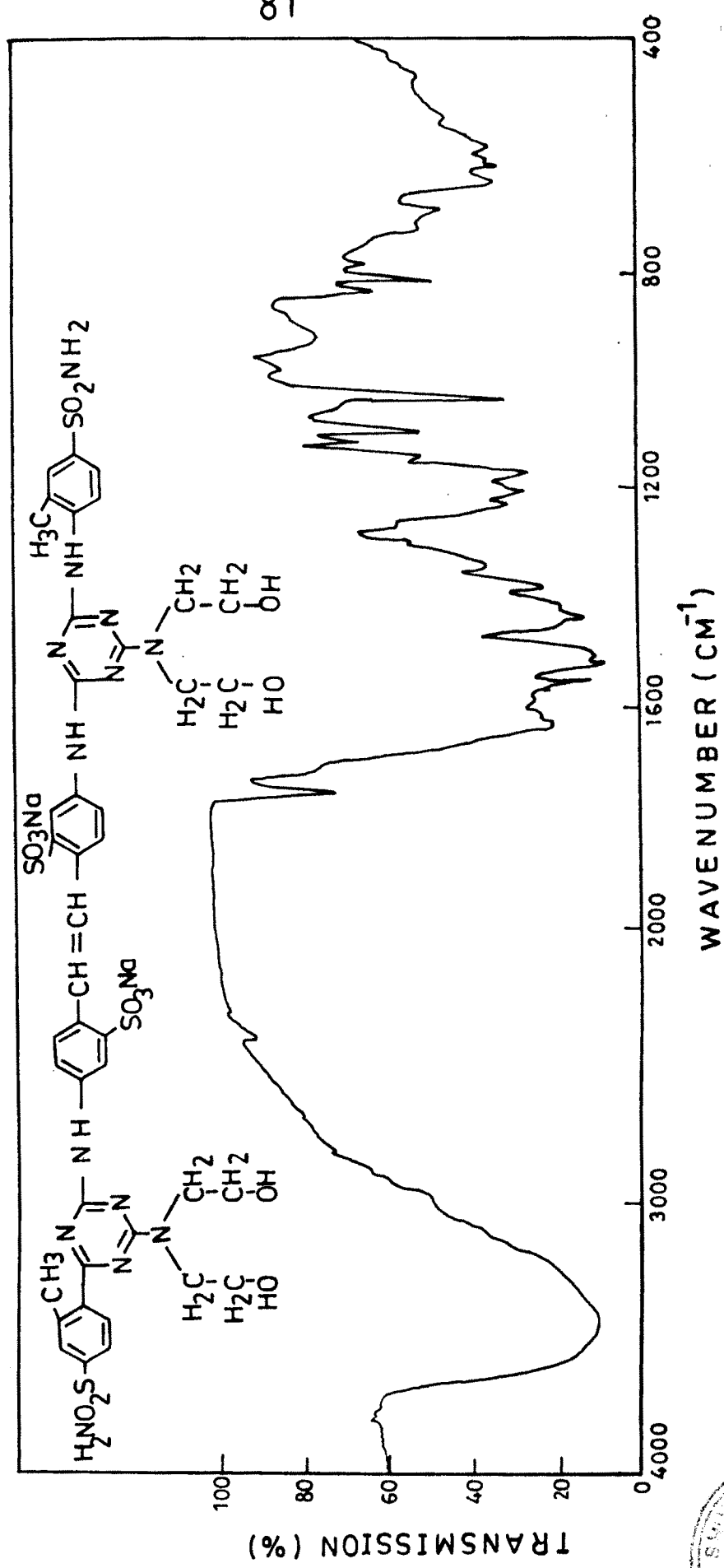


FIG.NO.

IR SPECTRUM OF 4,4' BIS [6(o-TOLUIDINE SULPHONAMIDO)-4-DIETHANOLAMINO
 1,3,5 TRIAZIN-2-YL] AMINO STILBENE 2,2' DISULPHONIC ACID.



TRANSMISSION (%)

WAVENUMBER (CM⁻¹)

FIG. NO.

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 RAJAWATI UNIVERSITY, KOLKATA



SYNTHESIS OF FLUORESCENT BRIGHTENER NO 3STEP 1- SYNTHESIS OF 6-(P-TOLUIDINE SULPHONAMIDO)-
2,4 DICHLORO -1,3,5 TRIAZINE

P Toluidine sulphonamide (9.3 gms , 2 mole) was dissolved in 0.01 M sodium hydroxide solution. (PH - 11). It was cooled in icebath at 0-5⁰C. Beaker was equipped with mechanical stirrer. Cyanuric chloride (10 gms , 2 mole) was dissolved in aqueous acetone (1:3).25 ml and then added in small quantity at a time and reaction was stirred vigorously. Reaction was continued after adding all quantity of cyanuric chloride. The PH of solution quickly fell from 11 to 2.8 . The reaction mixture was stirred for one hour.

STEP 2 - SYNTHESIS OF 4,4' BIS [6-(P-TOLUIDINE
SULPHONAMIDO)-4-CHLORO- 1,3,5 TRIAZIN -2-YL] AMINO STILBENE
2,2' DISULPHONIC ACID.

A paste of 4,4' diaminostilbene 2,2' disulphonic acid (8 gms , 1 mole) was added in above reaction mixture at room temperature. Addition was made slowly and dropwise. Condensation of DASDSA with cyanuric chloride required 2-3 hours. second chlorine atom was replaced at 35-40⁰C. To ensure reaction was completed. [condensation of primary amine with cyanuric chloride] was checked by testing unreacted aromatic primary amine by diazotization method.

STEP 3- SYNTHESIS OF 4,4' BIS [6(P-TOLUIDINE
SULPHONAMIDO) -4- DIETHANOL AMINO -1,3,5 TRIAZIN -2 -YL)
AMINOSTILBENE -2,2' DISULPHONIC ACID.

Reaction mixture was heated to raise the temperature 85-90⁰C. Diethanolamine (~~6.5gms~~ 2 mole) was added dropwise. Stirring was continued for 30 - 40 minutes. (P^H - 8.9) . Reaction mixture was cooled. It was salted out with sodium chloride and filtered. Washed with acetone, dried and the crude product was weighed.

Yield - 11 gms

M.P. - Decomposes on heating above 300⁰

Analytical data	Percentage of elements		
	C	H	N
Found	43.95	4.18	17.0
calculated	43.97	4.19	17.1

Spectra analysis :

1) U.V. A= 270 nm B= 360 nm.

2) I.R. NH (stretching) 3400 cm⁻¹

C= N(stretching) 1500 - 1600 cm⁻¹

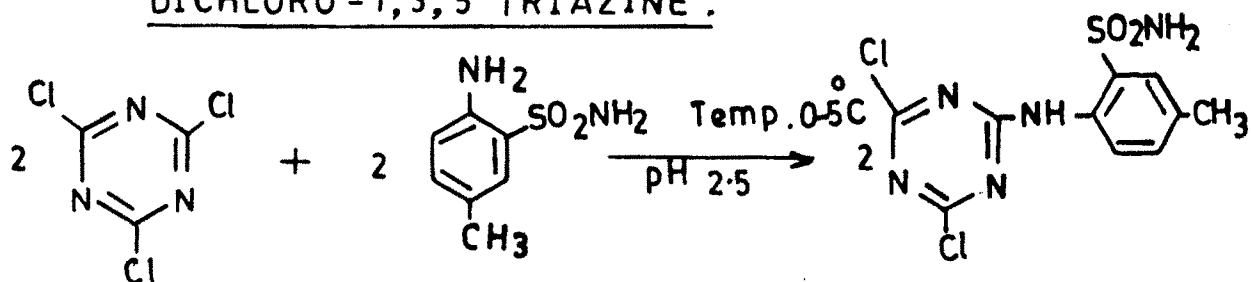
S= O(stretching) - 1160 cm⁻¹

S= O(stretching) - 1340 cm⁻¹

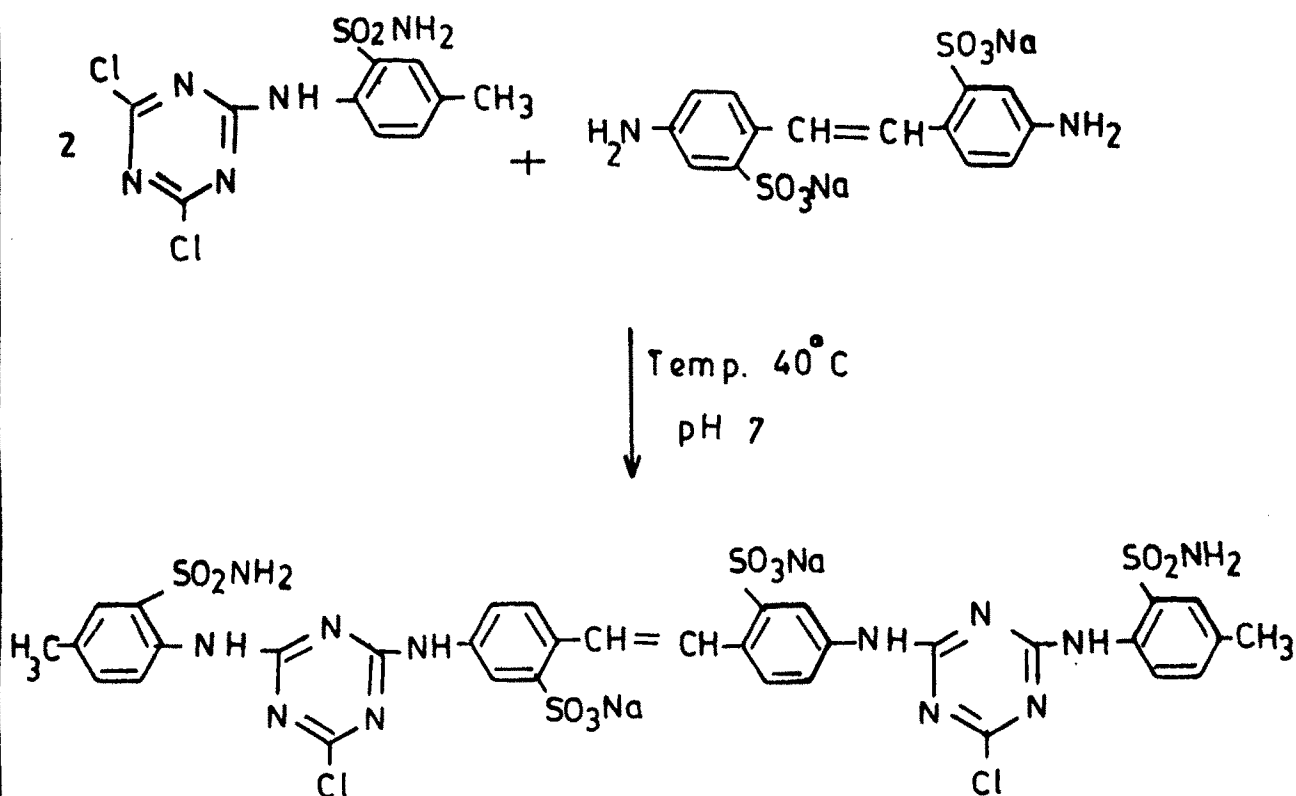
C= C(stretching) - 1600 - 1650 cm⁻¹

REACTIONS OF FLUORESCENT BRIGHTENER NO.3

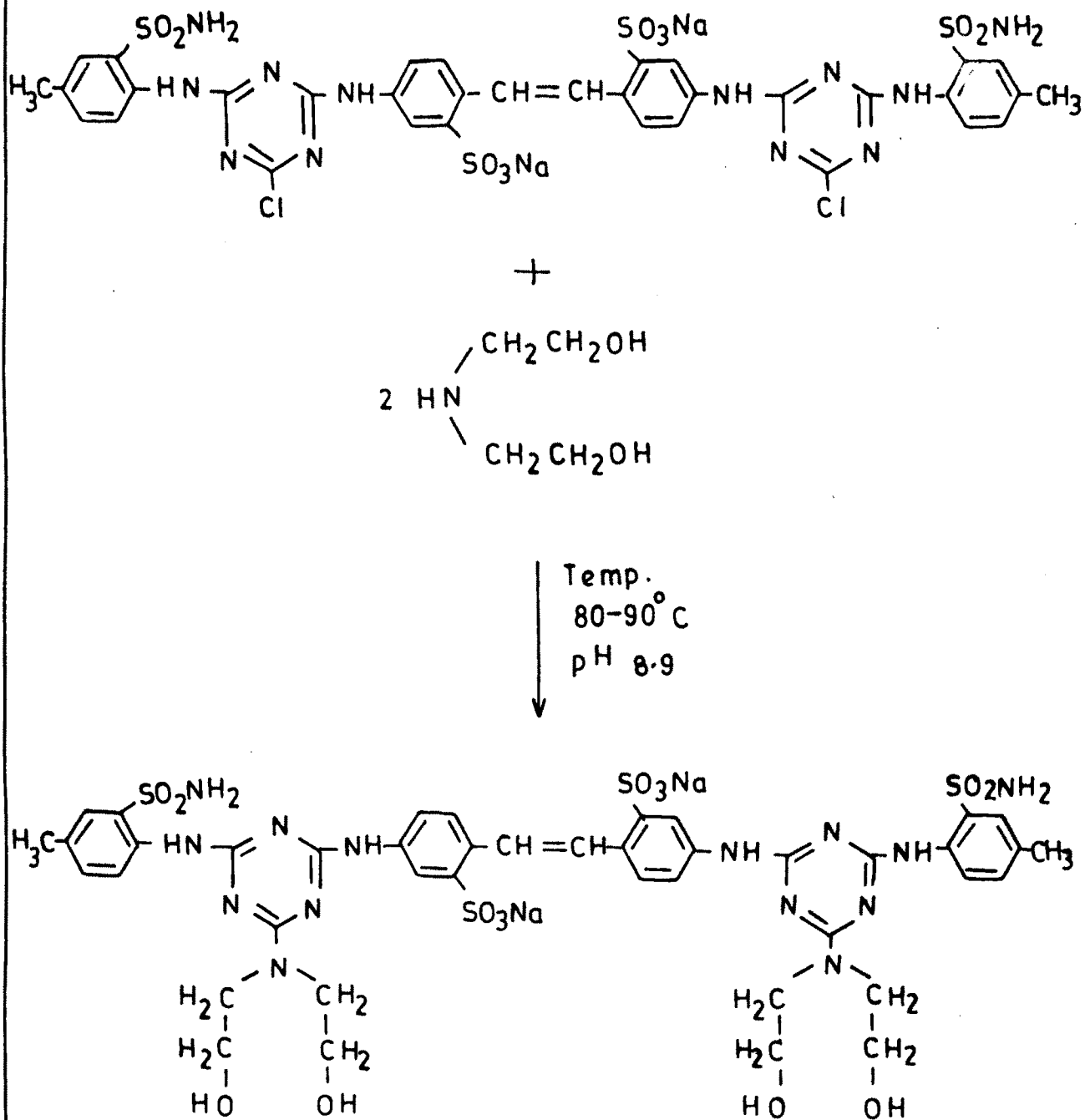
STEP I - SYNTHESIS OF 6 (P-TOLUIDINE SULPHONAMIDO 2,4
DICHLORO-1,3,5 TRIAZINE .



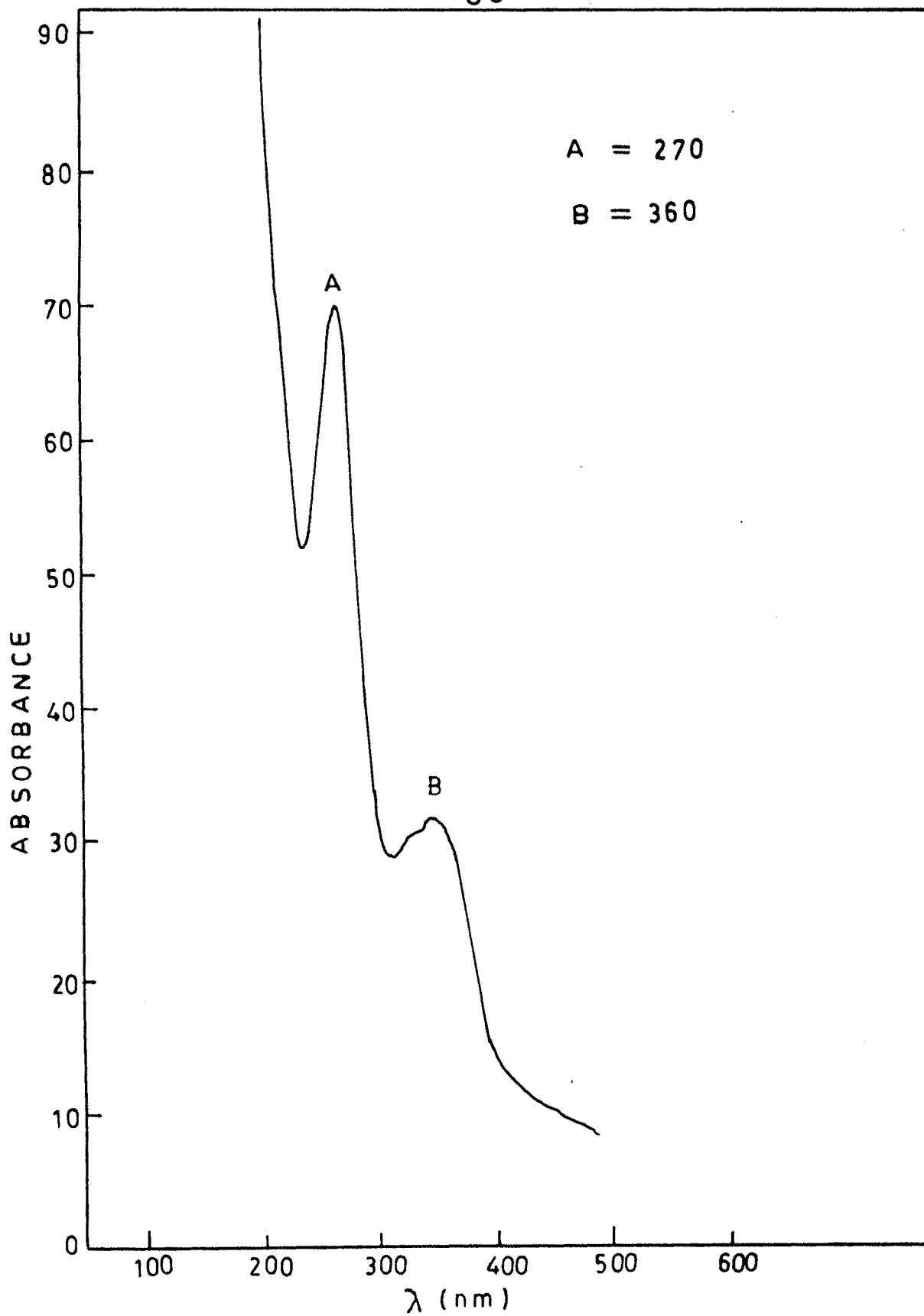
STEP II - SYNTHESIS OF 4,4' BIS [6 (P-TOLUIDINE SULPHONAMIDO)
-4-CHLORO-1,3,5-TRIAZIN-2-YL] AMINO STILBENE 2,2'
DISULPHONIC ACID .



STEP III - SYNTHESIS OF 4,4' BIS [6(p-TOLUIDINE SULPHONA-
-MIDO)-4-DIETHANOLAMINO-1,3,5 TRIAZIN-2 YL]
AMINO STILBENE 2,2' DISULPHONIC ACID .



FLUORESCENT BRIGHTENER NO.3



UV ABSORPTION SPECTRUM OF F. B. A. NO. 3

IR SPECTRUM OF 6 P-TOLUIDINE SULPHONAMIDO-2,4-DICHLORO-1,3,5-TRIAZINE .

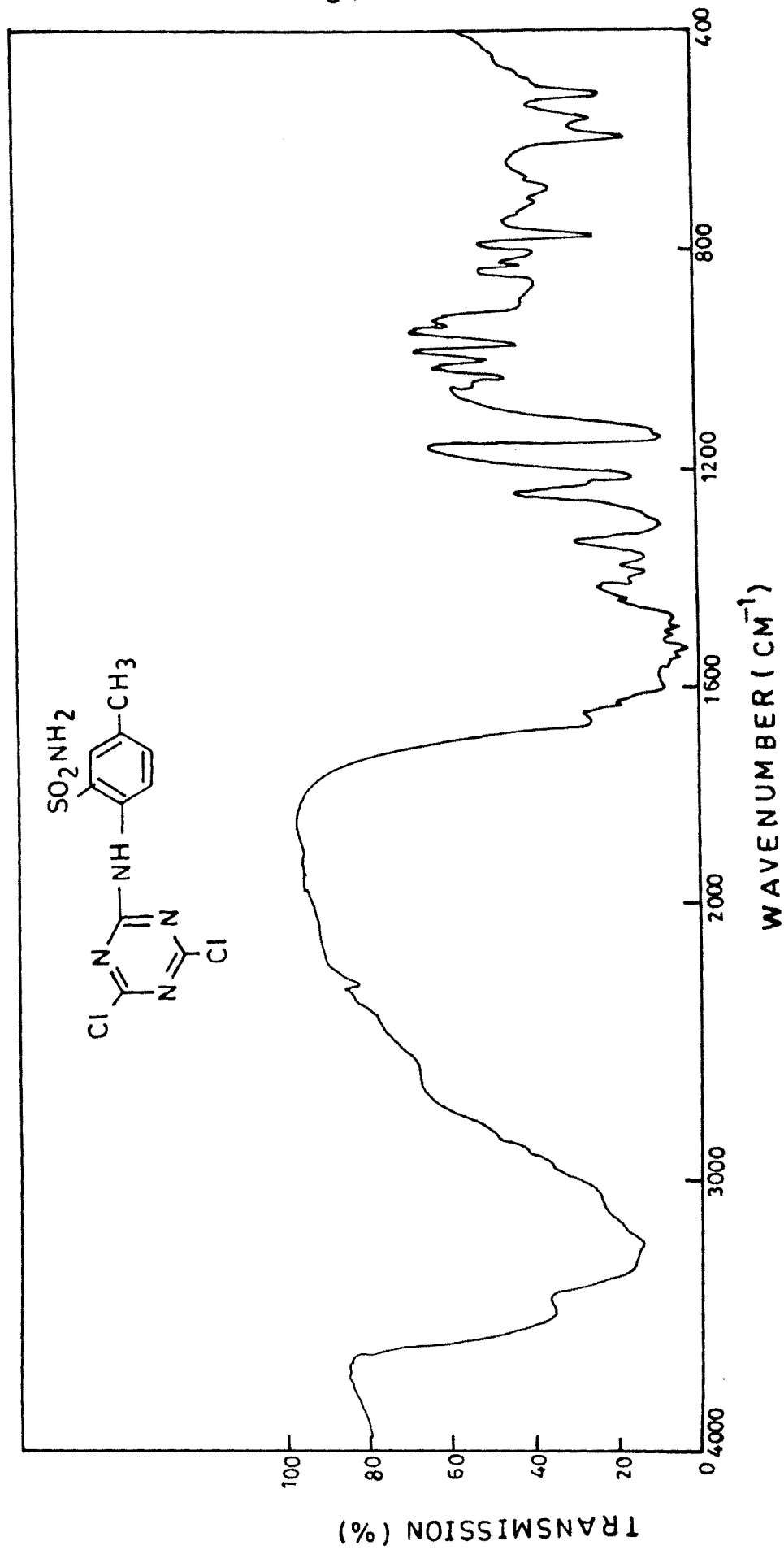


FIG. NO .

IR SPECTRUM OF 4,4' BIS [6 (P-TOLUIDINE SULPHONAMIDO)-4 CHLORO-1,3,5 TRIAZIN-2 YL]
 AMINO STILBENE -2,2'- DISULPHONIC ACID .

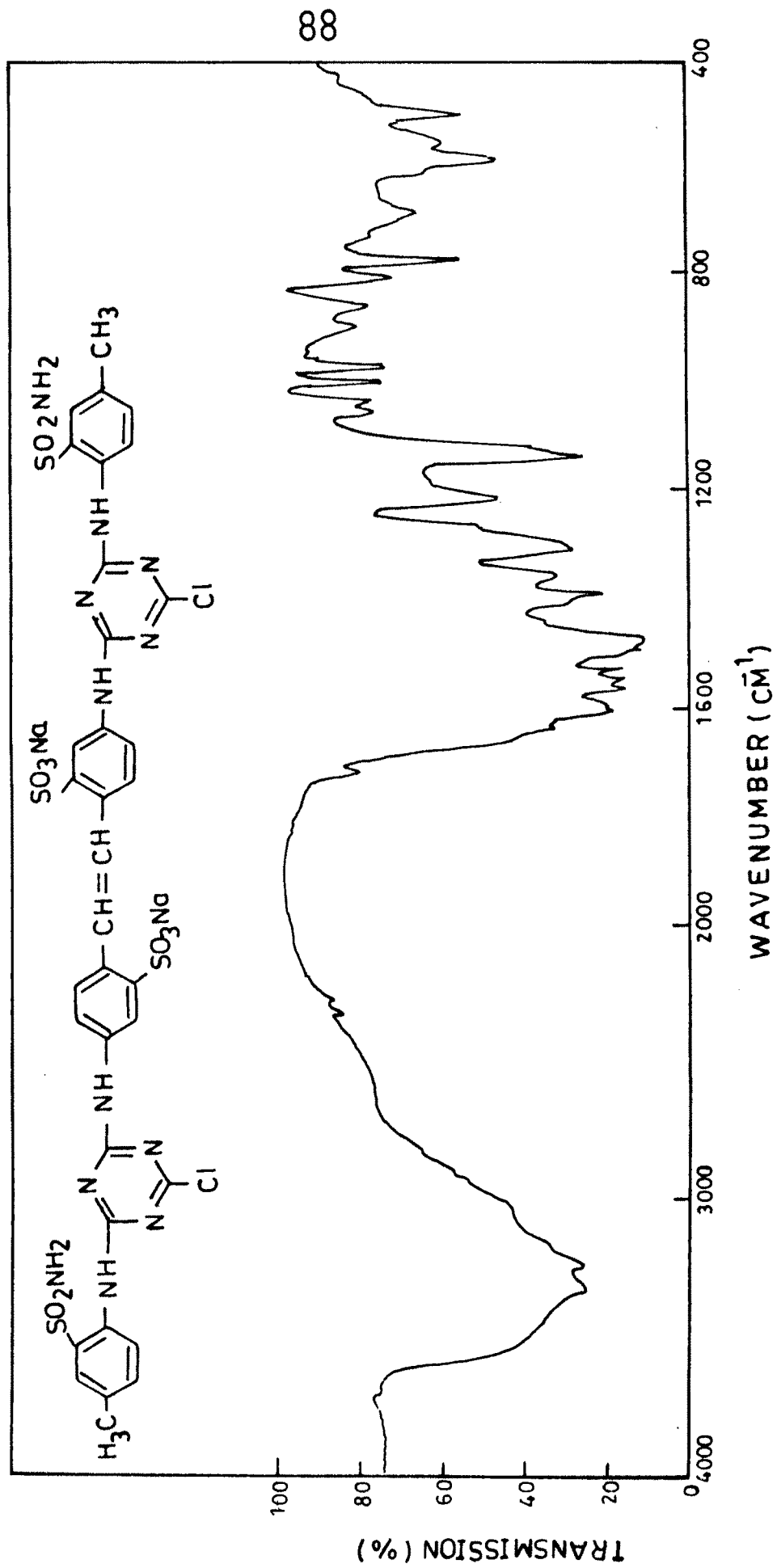


FIG. NO.

IR SPECTRUM OF 4,4' BIS [6-(p-TOLUIDINE SULPHONAMIDO) -4-DIETHANOL AMINO -
 1,3,5 TRIAZIN-2-YL] AMINO STILBENE-2,2'-DISULPHONIC ACID .

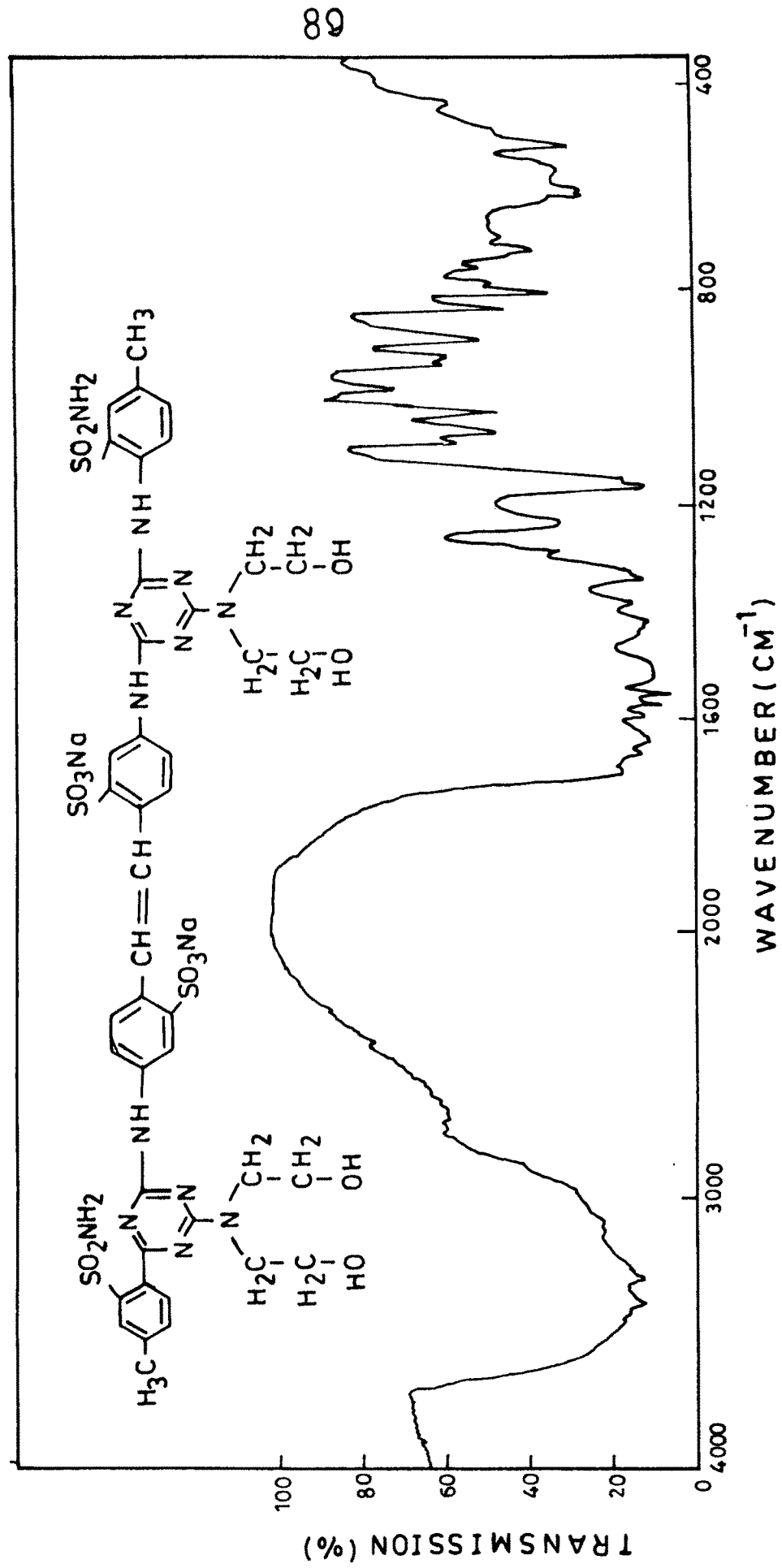


FIG. NO.

SYNTHESIS OF FLUORESCENT BRIGHTNER NO 4.STEP 1- SYNTHESIS OF 6(O-CHLOROANILINO)-2,4 DICHLORO-1,3,5 TRIAZINE.

Cyanuric chloride (10 gms . 2 mole) was dissolved in aqueous acetone (1:3) 25 ml .maintaining temperature at 0 - 5⁰C . Beaker was equipped with mechanical stirrer. O-chloroaniline (6.9 gms . 2 mole) was added in small quantity at a time and reaction mixture was stirred vigorously. Reaction was continued after adding all quantity of O-chloroaniline (PH - 4.5) . The reaction mixture was stirred for an hour.

STEP 2 - SYNTHESIS OF 4,4' BIS [6 - (O-CHLOROANILINO) -4- CHLORO - 1,3,5 TRIAZIN -2-YL] AMINO STILBENE - 2,2' DISULPHONIC ACID.

A paste of sodium salt of 4,4' diaminostilbene 2,2' disulphonic acid (8 gms . 1 mole) was added to the above reaction product at room temperature. Addition was made slowly and dropwise with continuous stirring. The completion of reaction [condensation of primary amine with cyanuric chloride] was checked by testing unreacted aromatic primary amine by diazotization method.

STEP 3- SYNTHESIS OF 4,4' BIS [6 (O-CHLORO ANILINO) 4 DIETHANOL AMINO 1,3,5 TRIAZIN- 2-YL] AMINO STILBENE -2,2' DISULPHONIC ACID.

Reaction mixture was heated to 85 - 90⁰C.
 Diethanolamine (6.5 gms, 2 mole) was added dropwise.
 Strring was continued for 30 - 40 minutes (p^H - 9.5).
 Reaction mixture was cooled. It was salted out with sodium
 chloride and filtered. washed with acetone, dried and the
 crude product was weighed.

Yield - 12 gms
 M.P. - Decomposes at 280⁰C

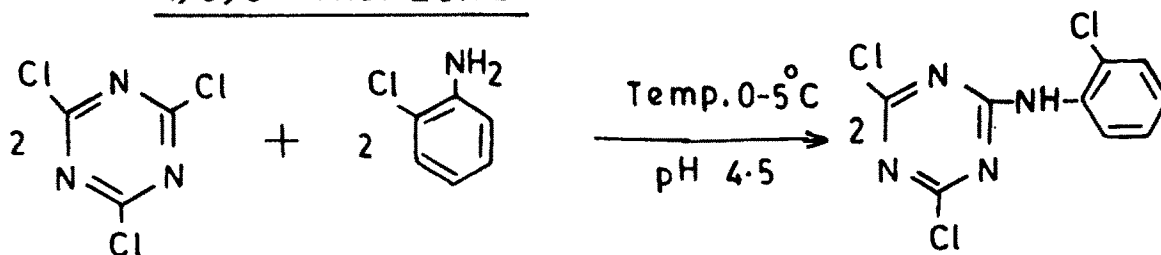
Analytical data	Percentage of element		
	C	H	N
Found	46.63	3.88	16.30
calculated	46.65	3.89	16.33

Spectral data

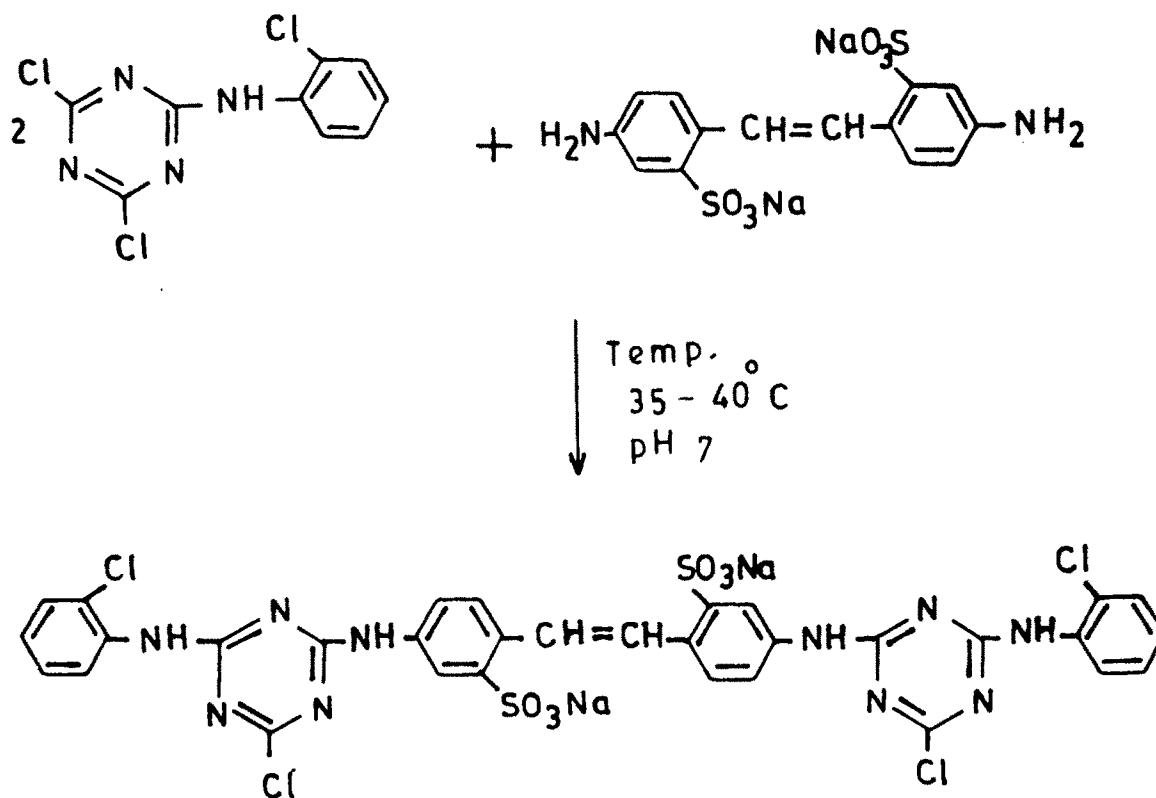
1) U.V. A= 220 nm. b= 350 nm
 2) I.R. NH(streching) -3400 cm⁻¹
 NH(bending) -1600 cm⁻¹
 C= C (stretching) 1500 - 1600 cm⁻¹
 C - C1 760 cm⁻¹
 C= C(streching) - ~~1600-1650~~ cm⁻¹
 1500 - 1600 cm⁻¹

REACTIONS OF FLUORESCENT BRIGHTNER NO.4

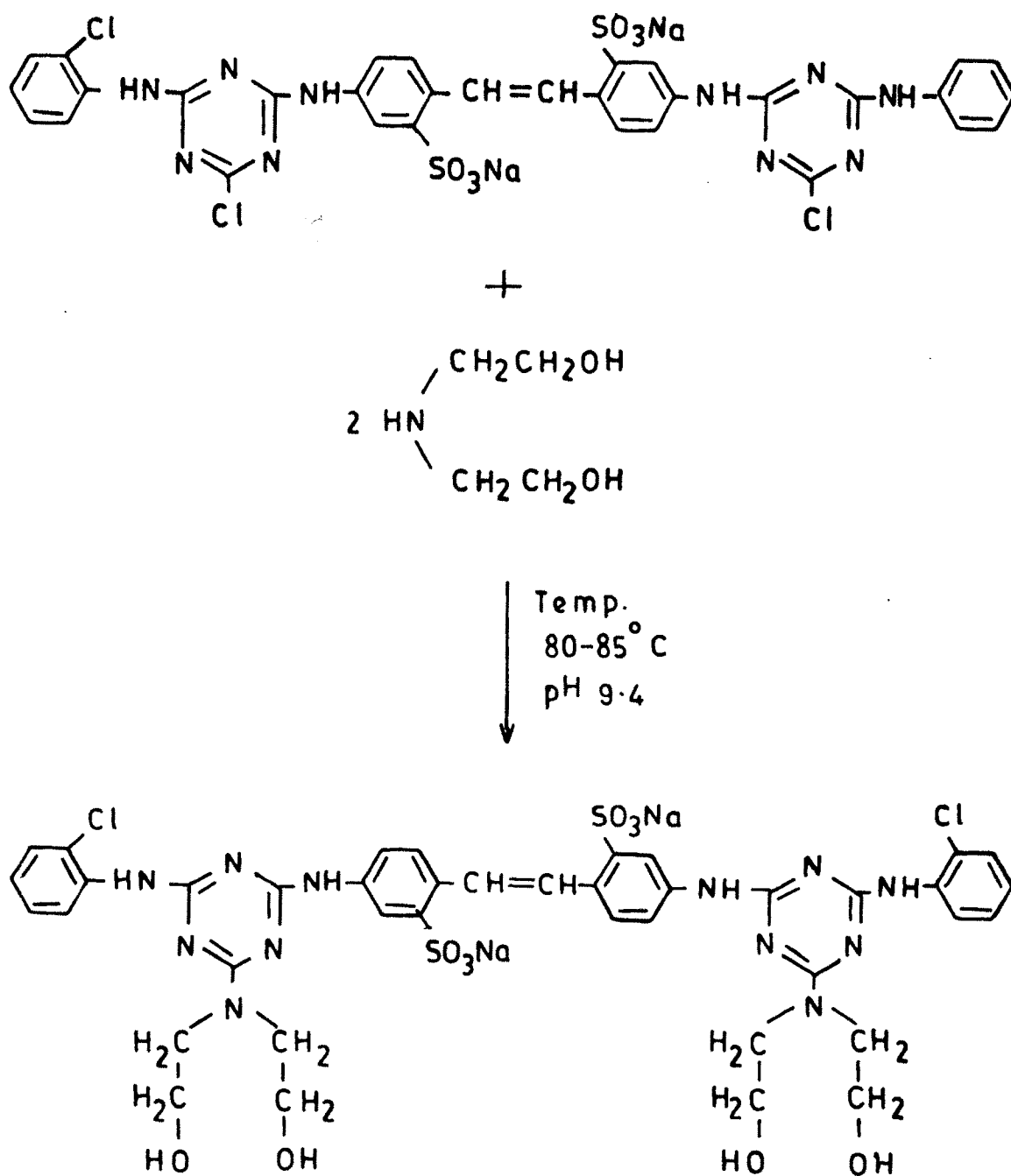
STEP I : SYNTHESIS OF 6 (o-CHLORO ANILINO)-2,4 DICHLORO-1,3,5 - TRIAZINE .



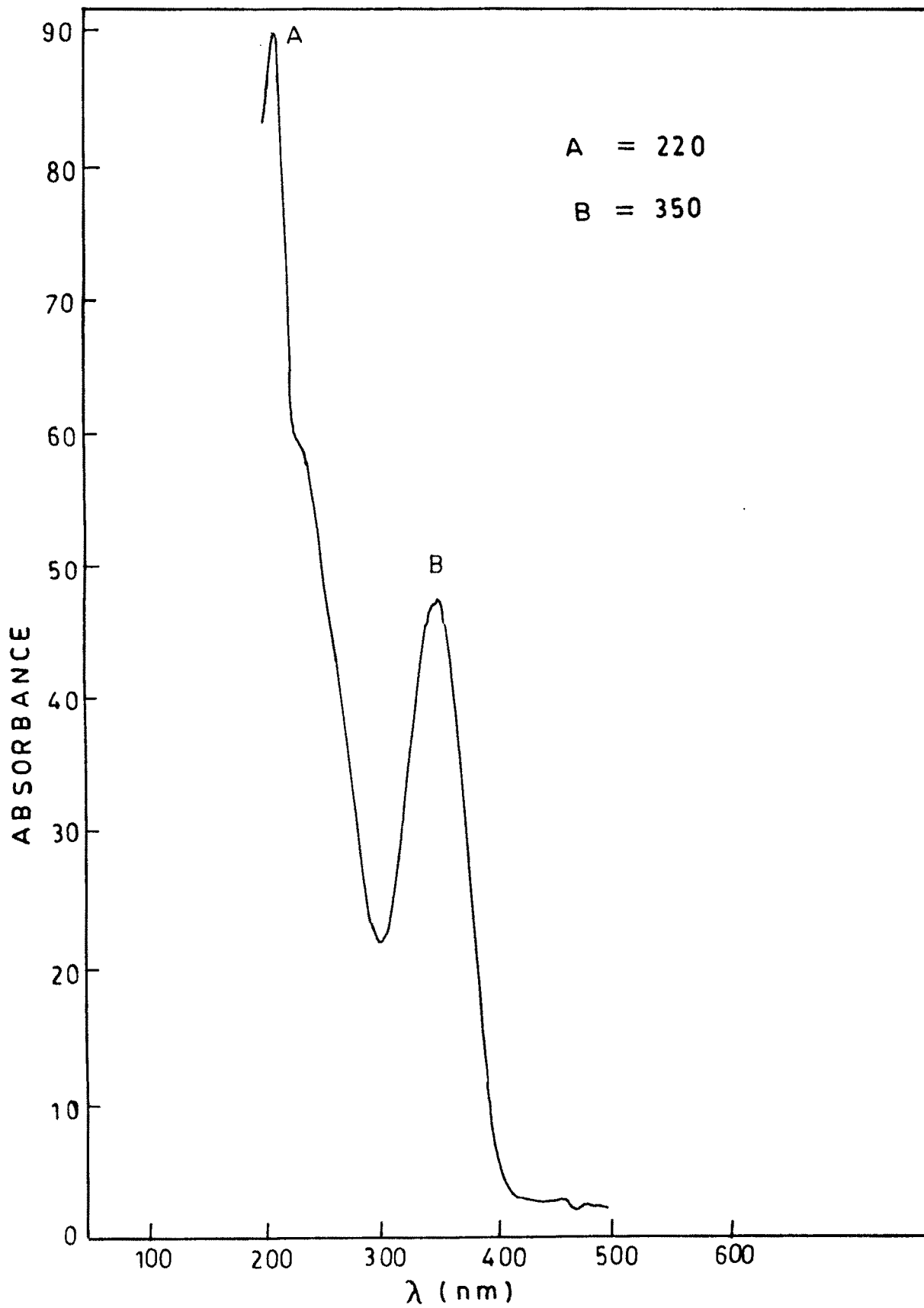
STEP II : SYNTHESIS OF 4,4' BIS [6 (o-CHLORO ANILINO)-4 - CHLORO 1,3,5 - TRIAZIN-2-YL] AMINO STILBENE 2,2' DISULPHONIC ACID .



STEP III — SYNTHESIS OF 4,4' BIS [6 (o-CHLORO ANILINO)-4-DIETHANOLAMINO-1,3,5 TRIAZIN-2-YL] AMINO STILBENE 2,2' DISULPHONIC ACID .



FLUORESCENT BRIGHTENER NO-4



UV ABSORPTION SPECTRUM OF F.B.A. NO. 4

IR SPECTRUM OF 4,4' BIS [6 (o-CHLOROANILINO) - 4 - DIETHANOLAMINO - 1,3,5 -
 -TRIAZIN-2-YL] AMINOSTILBENE - 2,2' - DISULPHONIC ACID .

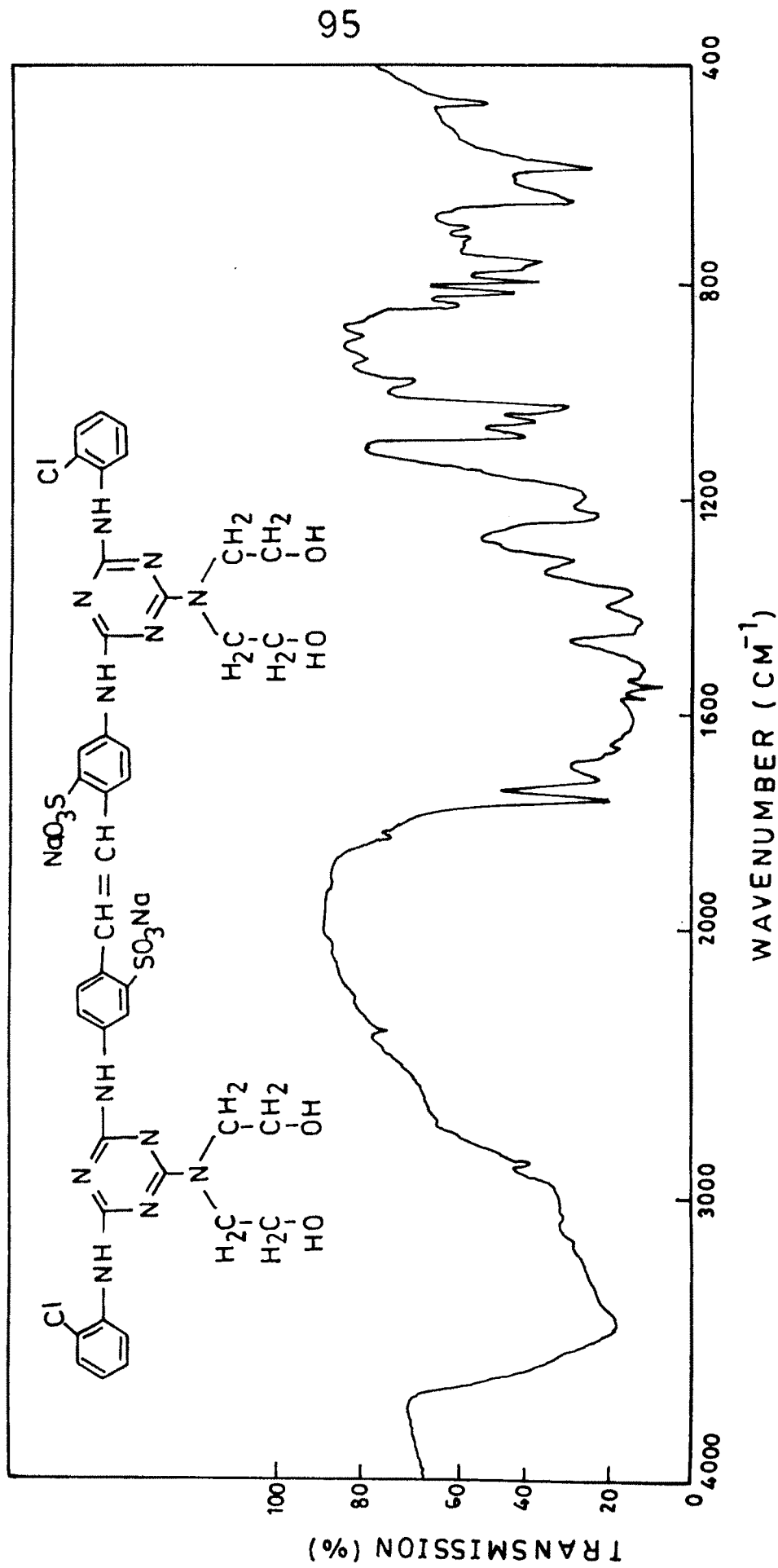


FIG. NO.

SYNTHESIS OF FLUORESCENT BRIGHTENER NO 5

STEP I- SYNTHESIS of 6 (1-NAPHTHYL AMINO)-2,4

DICHLORO - 1,3,5 TRIAZINE

Cyanuric chloride (10gms, 2mole) was dissolved in aqueous acetone (1:3), 25ml. maintaining temperature at 0-5⁰C.

Beaker was equipped with mechanical stirrer. 1 naphthyl amine (7.68gms, 2mole) was added in small quantity at a time and reaction was stirred vigorously. Reaction was continued after adding all quantity of 1-naphthyl amine (P^H 4.5). The reaction mixture was stirred for an hour.

STEP II- SYNTHESIS of 4,4' BIS [6-(1-NAPHTHYL AMINO)-4-CHLORO-1,3,5 TRIAZIN-2-YL] AMINO STILBENE. 2,2' DISULPHONIC ACID.

A paste of sodium salt of 4,4'-diaminostilbene 2,2'-disulphonic acid (8gms. 1mole) was added to the above reaction product at room temperature. Addition was made slowly and dropwise with continuous stirring. The completion of reaction [condensation of primary amine with cyanuric chloride] was checked by testing unreacted aromatic primary amine by diazotization method.

STEP III- SYNTHESES OF 4,4' BIS [6-(1-NAPHTHYL AMINO)-4-CHLORO-1,3,5 TRIAZIN-2, YL] AMINO STILBENE-2,2' DISULPHONIC ACID. Reaction mixture was heated 85-90⁰C. Diethanol amine (6.54 gms (6ml, 2mole) was added dropwise. Stirring was continued for 30-40 minutes. (P^H 9.4). Reaction mixture was cooled. It was salted out with sodium chloride and filtered, washed with acetone, dried and the crude product was weighed.

Yield - 13 gms

M.P. - Decomposes at 240⁰ C

Analytical data : Percentage of elements

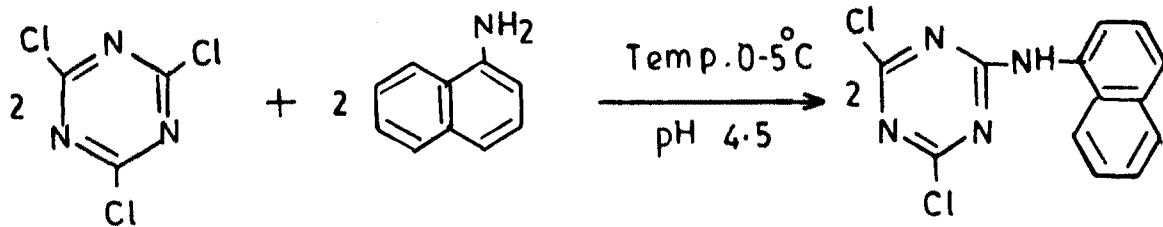
	C	H	N
Found	54.40	4.14	15.85
Calculated	54.44	4.16	15.88

Spectral data :

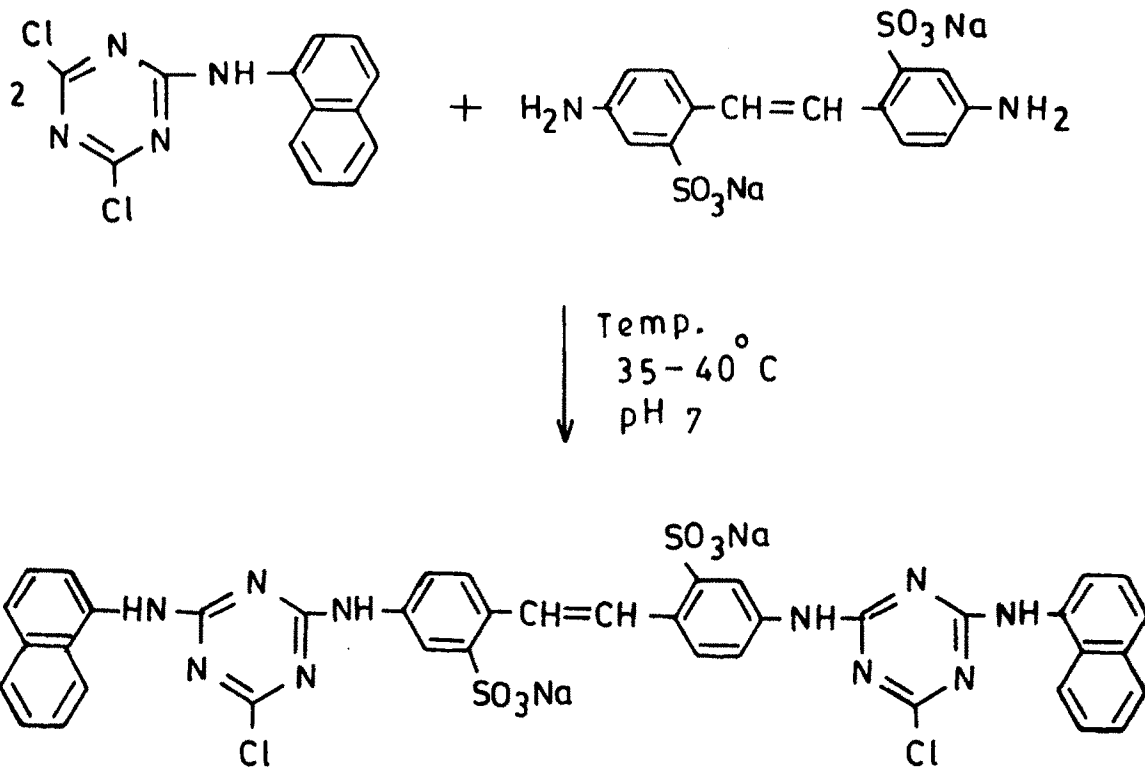
- 1) U.V. A = 225 n m B = 340 n m
- 2) I.R. NH (Stretching) 3400 cm^{-1}
- C=N (Stretching) 1500 1600 cm^{-1}
- NH (bending) - 1200 cm^{-1}
- C=C (stretching)- 1600-1650 cm^{-1}

REACTIONS OF FLUORESCENT BRIGHTENER NO. 5

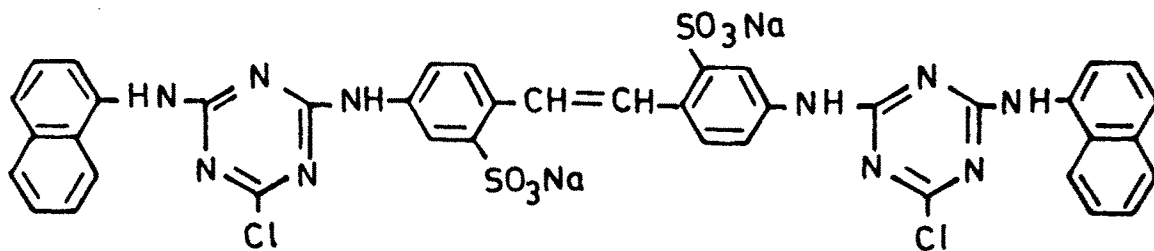
STEP I : SYNTHESIS OF 6-(1-NAPHTHYLAMINO)-2,4-DICHLORO-1,3,5-TRIAZINE.



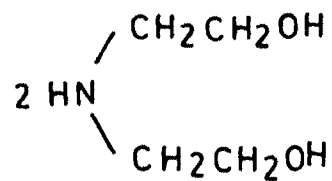
STEP II : SYNTHESIS OF 4,4'-BIS[6-(1-NAPHTHYLAMINO)-2-CHLORO-1,3,5-TRIAZIN-2-YL] AMINO STILBENE 2,2'-DISULPHONIC ACID.



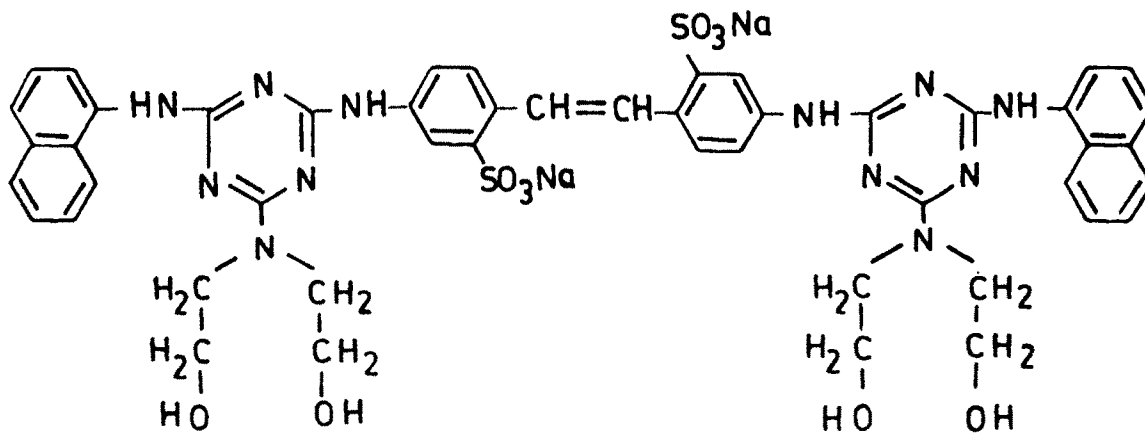
STEP III - SYNTHESIS OF 4,4' BIS [6-(1-NAPHTHYLAMINO)-4-DIETHANOLAMINO-1,3,5 TRIAZIN-2-YL] AMINO STILBENE-2,2' DISULPHONIC ACID .



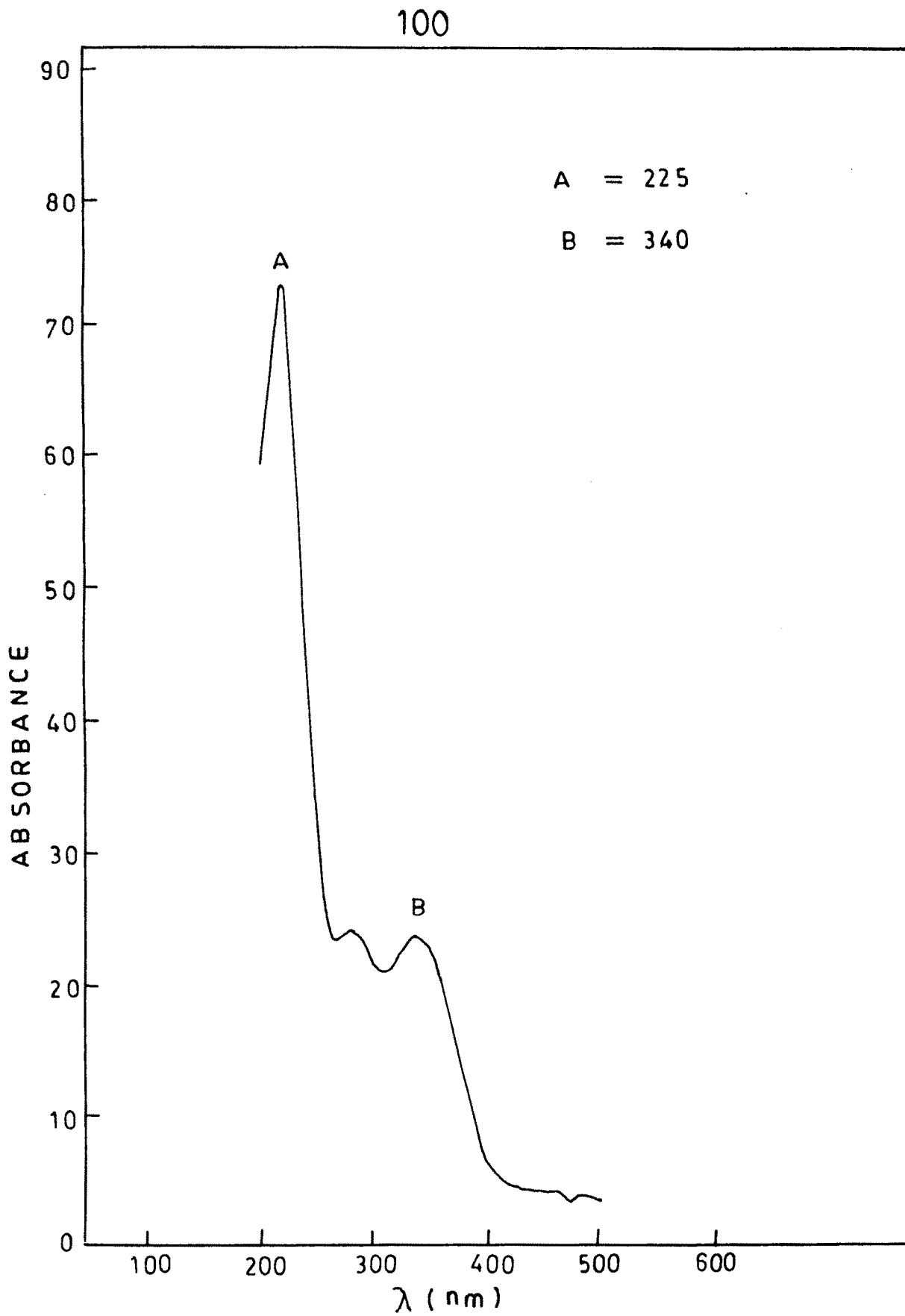
+



Temp.
80-85°C
pH 9.4



FLUORESCENT BRIGHTNER NO. 5



UV ABSORPTION SPECTRUM OF F.B.A. NO. 5

IR SPECTRUM OF 4,4' BIS [6-(1-NAPHTHYLAMINO)-4 DIETHANOLAMINO - 1,3,5 -
 TRIAZIN-2 YL] AMINOSTILBENE -2,2'- DISULPHONIC ACID .

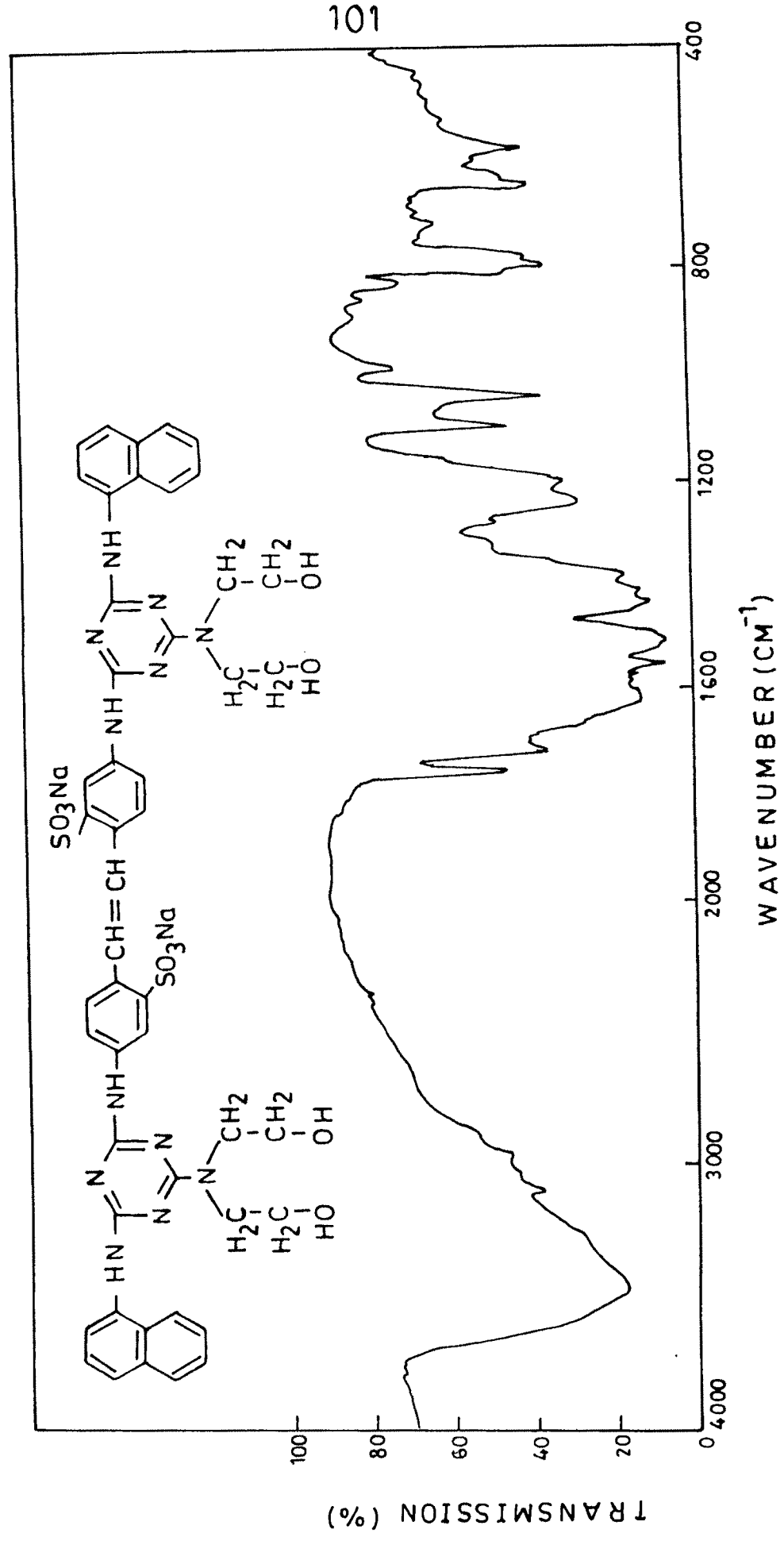


FIG. NO .