CHAPTER II

SYNTHESIS OF N-[7-CHLORO-4-QUINOLYL] SULFONAMIDS

2.1. INTRODUCTION

Many heterocyclic sulfonamides[1] show antibacterial activity against E. coli and S. aureus. Many quinoxaline[2] and quinoline sulfonimides[3,4] have been reported, to show antimicrobial activity against gram positive and gram negative bacteria.

2.2. PREVIOUS WORK :

Many quinoline derivatives including 4-substituted-7-chloroquinolines[5] and 8-hydroxyquinolines are known to exhibit microbial activity. These are used as antiameobic[6] drugs. Quinoline and Isoquinoline showed antifilarial properties and are used in treatment of worm infection.[7,8]

7-chloro-4-dialkylaminoquinoline show antimalarial activity.[9] 4-Amino-7-chloroquinoline with phenyl thiozole and phenyl dithiozole possess antibacterial and antiviral properties.[10] All the compounds of 4-amino-7-chloroquinoline series are tested for their antimalarial activity against 'plasmodium Burghei' in mice and antifilarial activity against "Lifomosoides cornii" in cotton rat and the results were negative.

(a) HALO-QUINOLINES

Haloderivatives of quinoline were used in the treatment of malaria.[11] They are prepared using different methods. Using "skruap quinoline synthesis[12] 2-and 4-haloquinolines

were synthesised. 2- and 4-Alkyl derivatives of quinoline are found active. Using Durbeyshire and Water[13] method chloroquinolines were prepared. According to this method, dry chlorine was passed at room temperature through the solution of quinoline in concentrated sulfuric acid containing silver sulfate. This gives a mixture of 5- and 8-chloroquinolines and 5,8-dichloroquinoline. These are used as antiamoebic[14] in man. 2-Quinoline on heating with mixture of phosphorus pentachloride and phosphorus oxychloride at 140°C gave 2-chloroquinoline[15]. 4,7-dichloroquinoline can be prepared by treating 7-chloro-4-hydroxyquinoline[16] with phosphorus oxychloride.

(b) HETEROCYCLIC SULFONAMIDES:

Following heterocyclic sulfonamides have been reported:

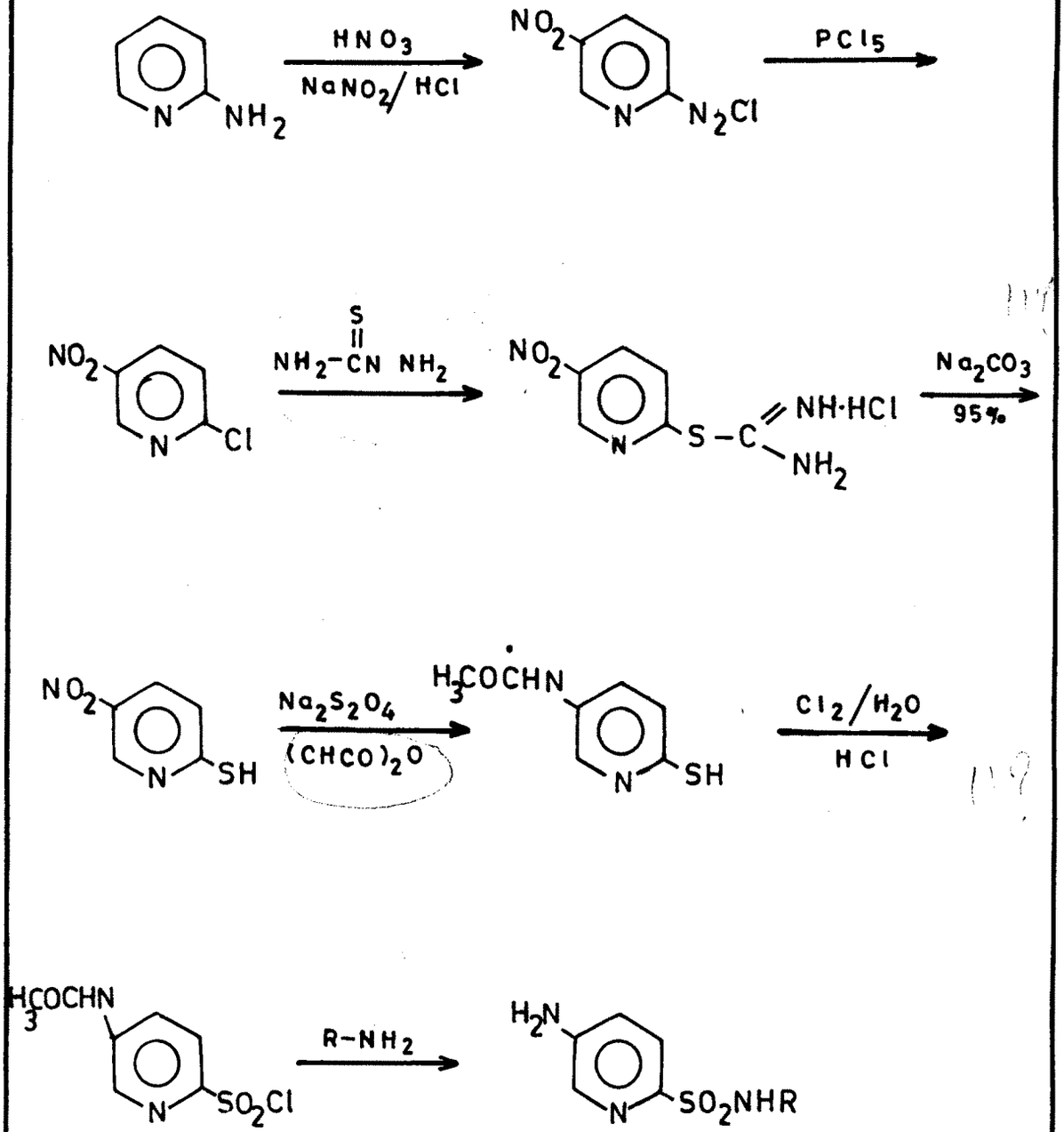
5-Amino pyridine-2-sulfonamides:

These compounds are prepared from 2-aminopyridine. 2-Aminopyridine was first nitrated, diazotised and converted to 5-nitropyridine. Them, this on treatment, with phosphorus pentachloride gave 2-chloro-5-nitropyridine. It was treated with thio-urea which gave 5-nitro 2-pyridyl-sendothiourea hydrochloride. This on hydrolysis with sodium hydroxide gave 2-mercapto-5-acetamidopyridine. On treatment with dithionit it was reduced to amine, then it on acetylation gave "2-mercapto-5-acetamido pyridine".

This on oxidation with chlorine in ice water was converted to 5-acetamide-2-pyridine sulfonyl chloride. This then underwent reaction similar to acetyl sulfanilyl chloride

CHART-I

Synthesis of 5-Aminopyridine-2-sulfonamide



in preparing, the desired sulfonamides[17] as shown in Chart I.

2-Amino-Pyridine-5-sulfonamides:

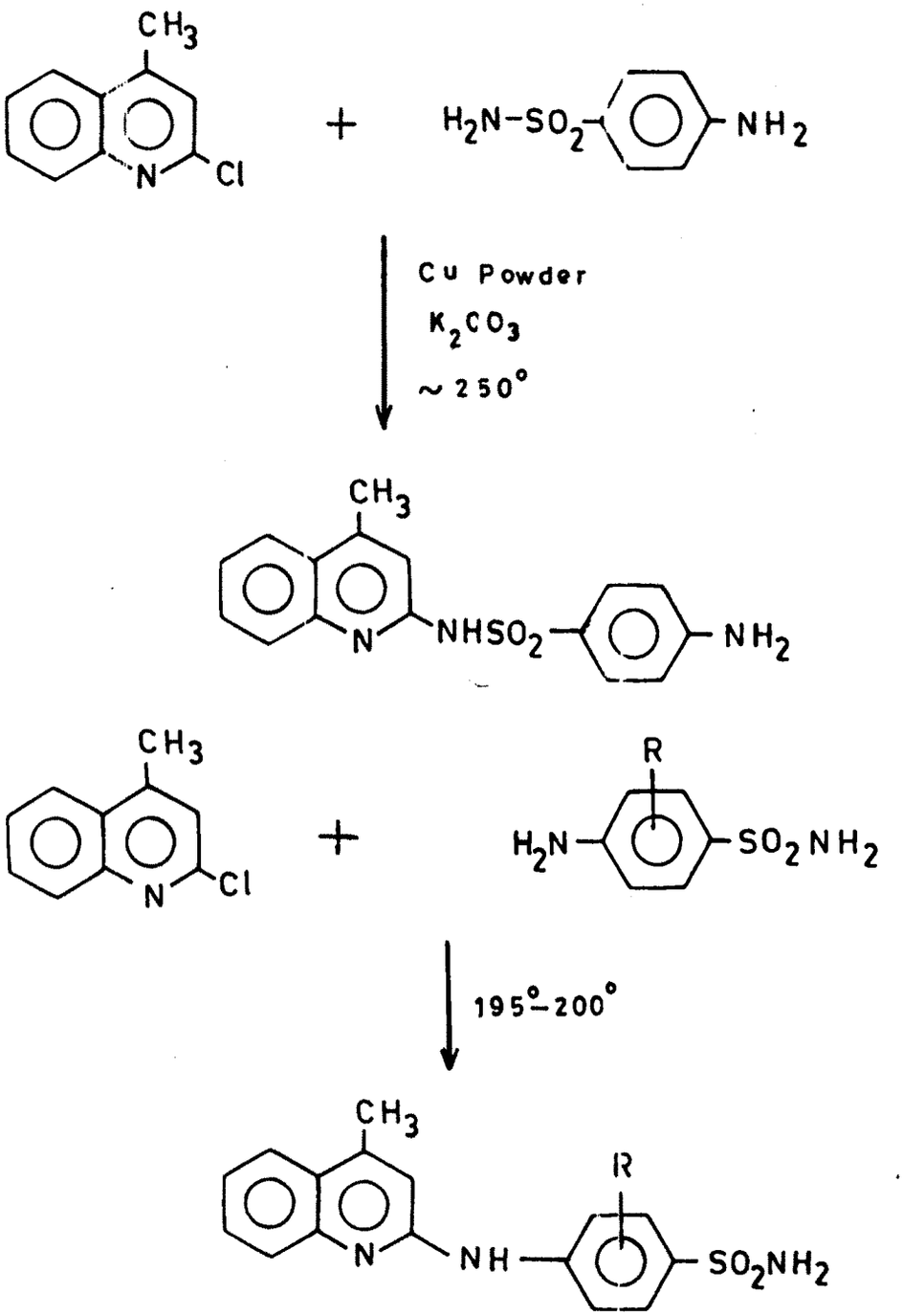
2 Amino-pyridine was directly sulfonated in 2-amino-5-sulfonic acid. It was diazotised and heated in water when it gave "2-pyridono-5-sulfonic acid". It on treatment with phosphorus pentachloride gave 2-chloroderivatives This on ammination gave "2-amino pyridine-5-sulfonamide".

Quinoline sulfonamides:

1) Recently quinoline sulfonamides i.e. N-(4-Methyl-2-quinolyl) sulfonamides and N (4-phenyl-2-quinolyl) sulfonamides were synthesised[3,4]. Aniline was refluxed with aceto acetic ester. The carbostyrol formed on treatment with phosphorus oxychloride gave 2-chloro-4-methyl quinoline. Then it was condensed with different substituted p-amino-benzene sulfonamides through - SO_2NH and -NH Linkages. About ten derivatives have been reported. All of them showed antibacterial activity against gram positive and gram negative bacteria. Scheme for synthesis of 2-chloro-4-methyl quinoline has been shown in Chart II.

CHART – II

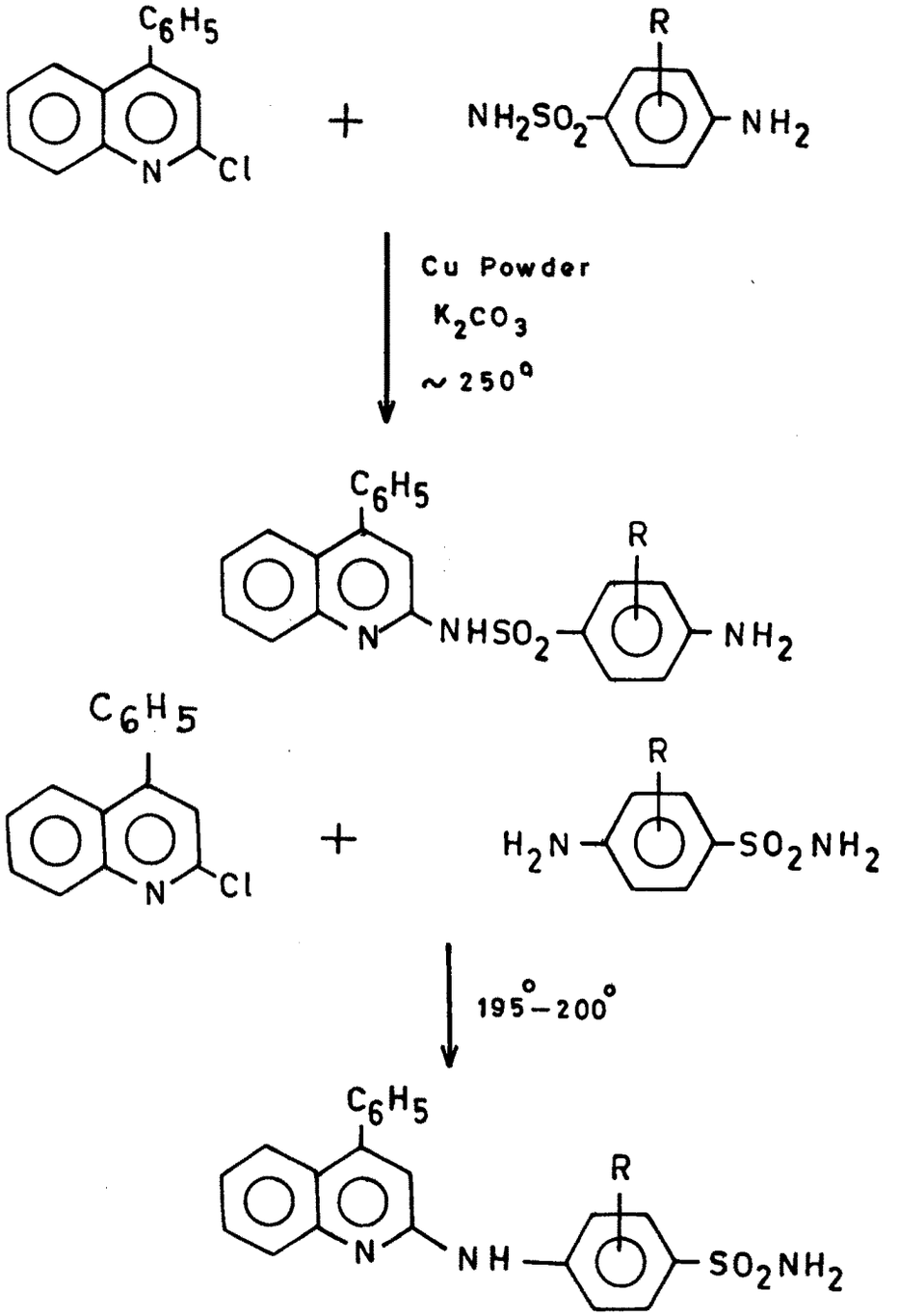
Synthesis of N[4-Methyl-2-quinolyl] sulfonamide.



ii) Recently N-(4-phenyl-2 quinolyl) sulfonamides[4] were synthesised. Aniline was refluxed with ethyl benzoyl acetate. When benzoyl acetanilide formed. Cyclization gave 4-phenyl-carbostyrol which when treated with phosphorus oxychloride gave 2-chloro-4-phenyl quinoline. Then it was condensed with different substituted aminobenzene sulfonamides through $-SO_2NH$ and -NH Linkages. About eight derivatives have been prepared. All of them showed antibacterial activity against gram positive and gram negative bacteria.

CHART-III

Synthesis of N[4-Phenyl-2-quinolyl]sulfonamide



2.3. PRESENT WORK

Discussion :

Heterocyclic sulfonamides exhibit an excellent microbial activity against S. aureus and E. coli. sulfonamide groups present in these compounds are responsible for their microbial activity. Similarly quinoxaline sulfonamides reported also show the antimicrobial activity. These are used in the treatment of tuberculosis.[18]

In chemotherapy, revealed that dialkylamine chin[19] plays a considerable part in the development of therapeutic activity. 4-Dialkylaminoquinoline shows excellent anti-malarial activity.[20] It has been also reported that 4-(-5-diethylamino 2' pentylamino-)-7-chloroquinoline[21] shows high antimalarial activity.

It was thought that, when substituted sulfonamides condensed with 4,7 dichloroquinoline they would form a series of good and useful antimicrobial drugs. With this view we prepared number of compounds by treating 4,7 dichloroquinoline with substituted sulfonamides.

4,7 Dichloroquinoline was prepared by using Price and Roberts[22] method as shown in Scheme I.

All N -7-chloro-4-Quinolyl-sulfonamide derivatives were prepared by treating-7-chloro-4 quinolyl moiety with variety of substituted sulfonamide derivatives through $-SO_2NH$ and -NH Linkage. These substituted sulfonamides were prepared by Method of C. Pellarino.[23]

The general outline of synthesis of substituted sulfonamides is shown in Scheme II.
Using this scheme sulfonamides prepared were

(1) p-Amino-benzene sulfonilamide

(2) p-Amino-Orthotoludine sulfonilamide

(3) p-Amino Meta toludine sulfonilamide.

Scheme II was used for attachment of 7-chloro-4 quinolyl moiety with substituted sulfonilamides through -NH linkage. This was carried by treating 4,7 dichloroquinoline[24] with substituted sulfonamides in presence of phenol at temperature 160°-165° for thirteen hours in paraffin bath. After completion the reaction mixture was cooled and poured into acid (4NHCl) and then made alkaline with NaOH solution. Solid product obtained, was recrystallised from benzene. Using this procedure the following sulfonamides were prepared.

1) 7-Chloro-4-(4'-sulfonamido-phenylimino) quinoline

1a) 7 Chloro-4-(4'-sulfonamido-2'-Methylphenylimino) quinoline.

7-Chloro-4-quinolyl sulfonamides through $-SO_2NH$ Linkage.

This was carried out by reacting 4,7-dichloroquinoline with p-aminobenzene sulfonilamides at temperature 250°C in paraffin bath in the presence of copper powder and potassium carbonate as catalyst. The progress of reaction was monitored by Thin Layer Chromatography (T.L.C.) at different intervals of time. All the reactions were completed within about 4 hours. The powdered product obtained was dissolved in water, acidified with 50% acetic acid, when solid was obtained. It was recrystallise from ethanol.

The following N 7 chloro-4-quinolyl sulfonamides were prepared by using scheme IV and V.

2) 7-chloro-4-(4'-amino 3'-Methylphenylsulfonimido)quinoline,

3) 7-Chloro-4-(4'-amino phenylsulfonimido) quinoline.

4) 7-Chloro-4-(4'-Amino -2' Methyl phenylsulfonimido) quinoline

5) 7-Chloro-4- (4'-Acetamido-phenylsulfanimido) quinoline

6) 7-Chloro-4 (4'-Acetamido-3' Methyl phenylsulfonimido) quinoline.

7) 7-Chloro-4-(4'-Methyl phenylsulfonimido) quinoline.

All these sulfonilamide derivatives are soluble in highly polar solvent such as alcohol, acetone and water. They are insoluble in chloroform, ether, pet ether etc. They have high melting points. Their proton magnetic resonance spectra have been taken in trifluoroacetic acid. These derivatives were tested for their antimicrobial activity against S. aureus, E. coli, B. substilis and Aspergillus niger. The Assay method has been used for testing their antimicrobial activity.

2.4. GENERAL REMARKS

(1) Structure and reactions are indicated by a double number, the first part of which indicates the chart to which it belongs and second part suggests the serial number of structure or reactions.

(2) Yield, percentage, physical constant, Molecular formula, elemental analysis of compound have been reported.

(3) Melting points are determined by open capillary method and are uncorrected.

(4) PMR spectra were recorded in CCl_4, $CDCl_3$ and TFA with tetramethyl silane (TMS) as an internal reference. The chemical shifts are in ppm.

(5) The purity of the compounds was checked by T.L.C. using silica gel, as adsorbent.

(6) I.R. spectra are recorded the values of stretching frequencies are in Cm^{-1}.

EXPERIMENTAL WORK

2.5 Preparation of 4,7-dichloroquinoline:[22]

(1) Preparation of ethyl α-carbethoxy β-m-chloroanilino-acrylate :

A mixture of 12.8 gm (0.1 mole) of m-chloroaniline and 23.3 gm (0.108 mole) of ethoxymethylene malonic ester was stirred until homogeneous, a few boiling chips were added and the flask was heated in oil bath at 100° for an hour. The reaction mixture prepared in these ways were normally used directly in the next step without further purifying the acrylate. M.P. = 55-56°.

(2) 3-Carbethoxy-7-chloro-4-Hydroxyquinoline :

The molten acrylate (0.1 mole at 100°) was poured slowly through the top of air condenser into 100 c.c. boiling diphenylether. Thirty minutes after the addition the cyclized product began to crystallise on the wall of flask. The crystals soon filling the boiling solution. The mixture was heated for total of 45 minutes and then allowed to cool room temperature to the semisolid mass was added to 50 c.c. of high boiling petroleum ether. The mixture was stirred well, the crystalline product was collected and washed on filter paper with two 50 c.c. portions of high boiling petroleum ether. The remainder of the solvent was removed by means of high boiling petroleum ether in a soxhlet extractor to yield

21 gm.(80%) of quinoline ester. M.P. = 295-297°.

3-Carbothoxy-7-chloro-4-hydroxyquinoline was very insoluble in water, alcohol, benzene, petroleum ether, chloroform and ethyl acetate, it was slightly soluble in acetic acid and more so in pyridine. Recrystallised from pyridine. M.P. = 295-297°.

(3) 7-Chloro-4-hydroxyquinoline-3-carboxylic acid :

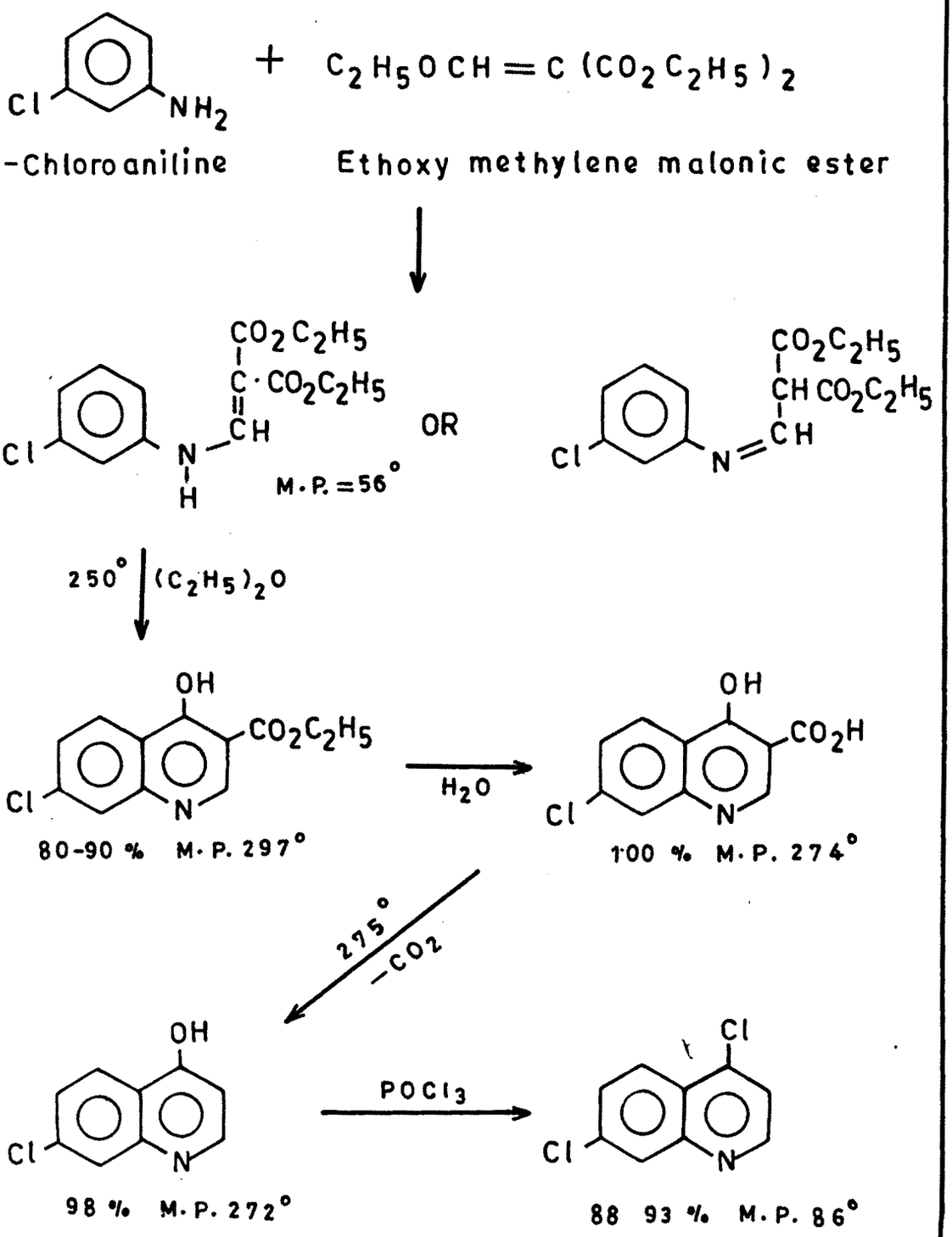
3-Carbethoxy-7-chloro-4-hydroxyquinoline (3.93 gm. C.0156 mole) was boiled under reflux with 10% NaOH solution (32 c.c.) for one hour. A few gms of decolourised charcoal was added and mixture was boiled for additional five minutes, filtered and washed with 20 c.c. of hot water. The filterate was acidified with 10% HCl and white 7-chloro-4 hydroxyquinoline 3-carboxylic acid was collected on filter paper and washed. After drying thoroughly in vacuum desiccator the product weighted 3.48 gm. The quinoline acid soluble in dil alkali and soluble in ethanol. M.P. = 273°-274°.

(4) 7-Chloro-4-Hydroxyquinoline :

In a 4 litre beaker take 7-chloro-4-hydroxy-3-carboxylic acid and decarboxylated by heating until effervescences ceased and then allowing the product to cool and solidify. The yield of crude 7-chloro-4-hydroxyquinoline is 98% of theoretical amount. The pure 4-hydroxyquinoline obtained by recrystallisation of crude decarboxylation product,

SCHEME-I

Synthesis of 4,7 dichloroquinoline.



from large amount of water after decolourisation with fine needles

M.P. = 270 - 272°

(5) <u>4,7 dichloroquinoline</u> :

The crude 7 chloro-4 hydroxy quinoline (34 gm) was refluxed for two hours with 110 c.c. of $POCl_3$. Most of $POCl_3$ removed under reduced pressure and residue was poured in to ice water. The mixture was made alkaline with NH_4OH and the product taken up in methylene chloride.

M.P. 84.5° - 85°

yield = 32 gm.

The above experimental work has been represented by Scheme I.

General procedure for the preparation of substitute p-amino benzene sulfonilamides[25]

Acetanilide was prepared by heating mixture of Aniline (20 ml.) glacial acetic acid (20 ml.) and acetic anhydride (21 ml.) and 0.1 gm. Zinc dust. It was poured into ice cold water. Product obtained was filtered and recrystallised from boiling water.

Yield 18 gms. m.p. = 114° (Lig. 114°)[20]

To above acetanilide (18 gms) Chlorosulfonic acid (90 gms) added in small quantity at a time, then heated for one hour. The oily solution obtained was added to ice cold water very carefully, when p-aminobenzyl sulfonyl chloride was formed. It was filtered.
Yield 16 gms. m.p. 146° (Lig 146°)[20]

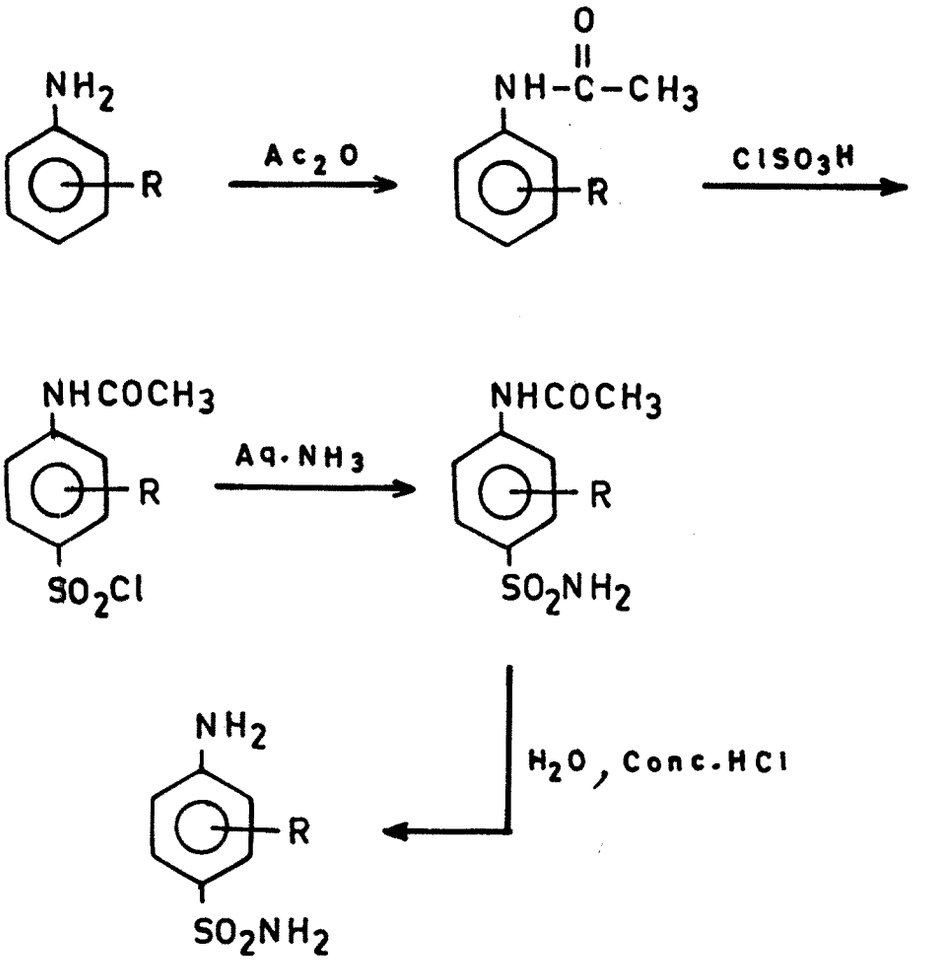
To above crude product (16 gms), concentrated ammonia (70 ml) and water (70 ml) were added and heated just below its boiling point for about fifteen minutes. Then dilute sulfuric acid was added till the mixture was acidic to congo red paper. p-Acetamido benzene sulfonilamide was precipitated. It was filtered and dried.

Yield 14 cms M.P. 218° (Lit. 218°)[20]

To above crude product (14 gms), hydrochloric acid (10 ml) and water (30 ml.) was boiled for about 30-45 minutes. It was cooled and filtered. Then charcoal (2 gms) was added

SCHEME-II

Preparation of p-amino benzene sulfonamide



Compounds :—

Ia R = H

IIb R = CH_3 ; ortho to NH_2

IIIc R = CH_3 ; meta to NH_2

to filtrate and it was boiled for few minutes. The solid sodium carbonate (16 gms) was added till solution was just neutral, when precipitate of p-aminobenzenesulfonilamide was obtained. It was recrystallised from ethanol.

Yield, 13 gms m.p. 161° (Lit 153°)[20]

By this procedure ortho toluidine sulfonilamide, meta toluidine sulfonilamide, meta toluidine sulfonilamide were prepared.

Scheme II represents synthesis of above compounds :

Synthesis of 7-chloro-4-(4' sulfanilamidophenyl-imino) quinoline.

(A) General procedure for attachment of quinolyl moiety through -NH Linkage :

Equimolecular quantities of 4,7-dichloroquinoline (0.500 gms) and substituted sulfonilamide (0.30 gms) and phenol (0.202 gm) gaken and heated for half an hour at 100° to 160° and for twelve hours at 160° to 165°. Then it was cooled and poured into solution of 60 ml. of 4 N HCl. The red brown solution was treated with Dacron and filtered. It was then made alkaline with NaOH solution (15 gm in 50 ml. water). A yellow oily layer was obtained which after stirring for 15 minutes solidified to white granular solid. The crude product recrystallised from benzene.

Scheme III represents Synthesis of these derivatives. Using this procedure following derivatives were prepared. Their melting points were found.

1) 7-chloro-4-(4'-sulfonamide phenyl imino) quinoline

M.P. = 198° Molecular formula : $C_{15}H_{12}N_3SO_2Cl$

		Practical	Theoretical
Elemental Analysis	C	53.85 %	53.97%
	H	3.38 %	3.59%
	N	12.47 %	12.59%

I.R. 3400, 1280 (S=O) cm^{-1}

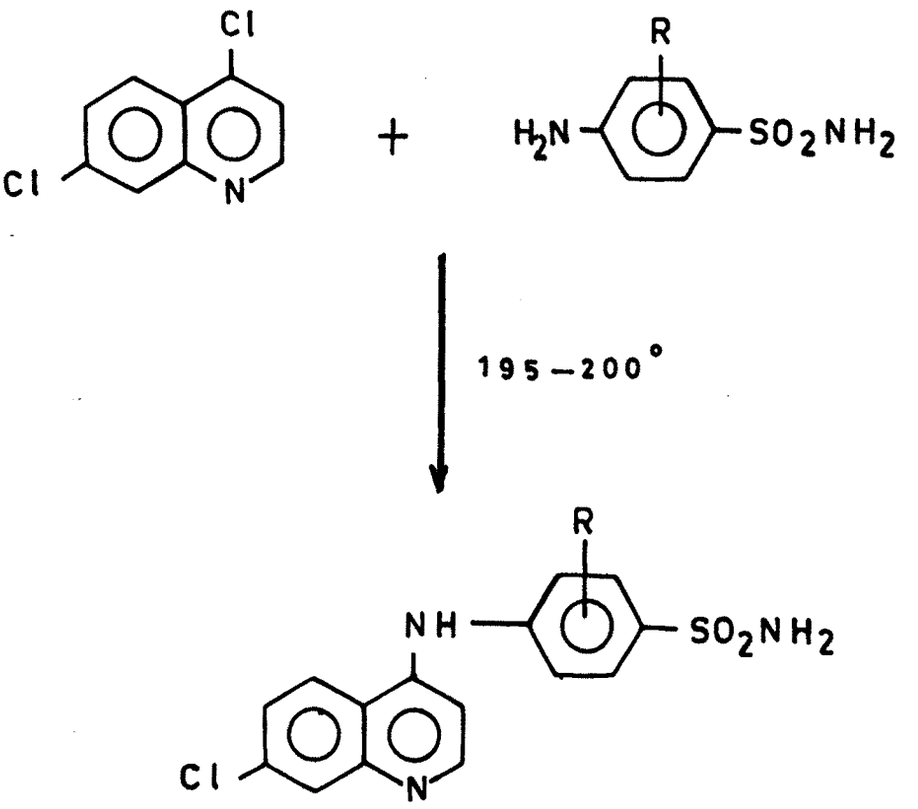
1 (a) 7-Chloro-4-(4'-sulfonamide-3'-Methyl phenyl imino) quinoline.

could not be obtained.

SCHEME-III

Synthesis of

7-Chloro-4-[4'-sulfonimido phenyl imino]quinoline



Compounds :—

(1) R = H

1(a) R = CH_3 ortho to SO_2NH_2

(B) General procedure for attachment of quinolyl moiety through - $NHSO_2$-Linkage :

4,7-dichloroquinoline (0.500 gm), substituted p-amino-benzene sulfonamide (0.400 gm) and anhydrous potassium carbonate (0.400 gm) and copper powder (0.05 gm) were heated for about four hours. The powdered product obtained was boiled with 0.2 N Sodium hydroxide (40 ml.) for about 10 minutes. Then it was acidified with dil acetic acid which gave gammy solid. This gummy solid was dissolved in 2 N Sodium hydroxide (20 ml.) and boiled for few minutes. The solution cooled and salty material formed was dissolved in water. Then it was treated with 50% acetic acid a solid compound obtained was filtered and their M.P. were found.

Schemes IV and V represent the synthesis of these compounds. Using this procedure following sulfonilamides were prepared.

(2) 7-Chloro-4-(-4'-Amino-3' methyl phenyl sulfonilamide) quinoline

Recrystallied from ethanol.

M.P. = 285° Mol. formula - $C_{16}H_{14}N_3O_2SCl$

Yield = 65 %

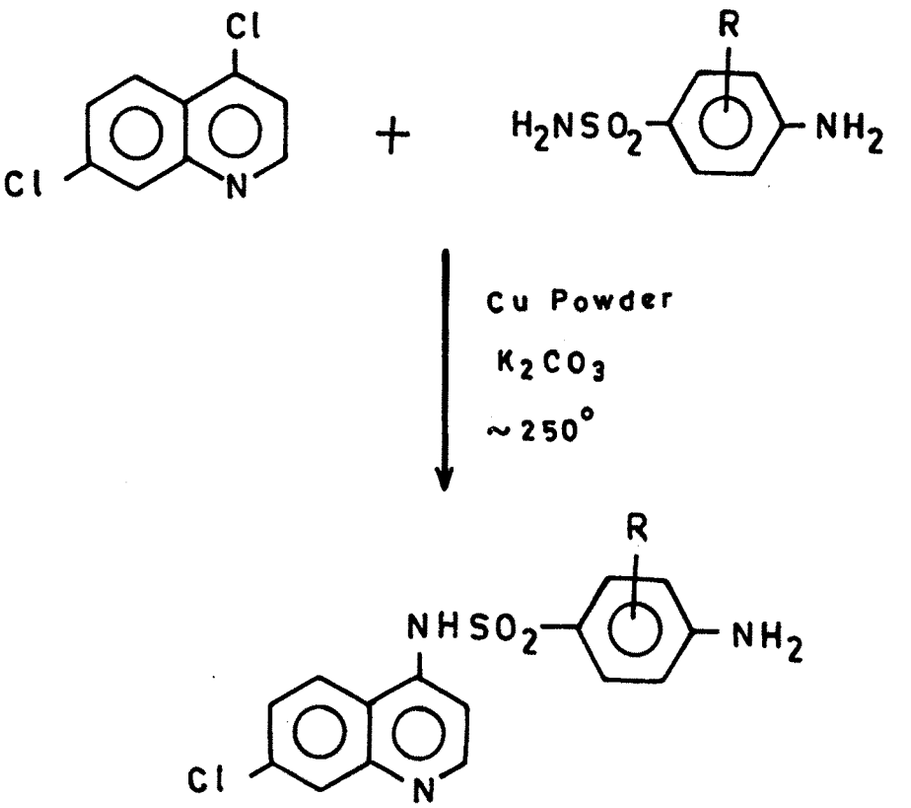
		Practical	Theoretical
Elemental Analysis	C	55.40 %	55.25 %
	H	3.98 %	4.02 %
	N	12.10 %	12.08 %

I.R. 3440, 3355 (NH, NH_2) 1555, 1350, 1160 (S = O)

SCHEME-IV

Synthesis of

7-Chloro-4-[4'-Amino phenyl sulfonimido]-quinoline



Compounds :—

2) R = CH_3 ortho to NH_2

3) R = H

4) R = CH_3 meta to NH_2

(3) 7-Chloro-4-(-4'-Aminophenyl sulfonimido) quinoline

M.P. = 300° Mol. formula. $C_{15}H_{12}N_3O_2ClS$

Yield 75 %

		Practical	Theoretical
Elemental Analysis	C	53.96 %	53.97 %
	H	3.58 %	3.59 %
	N	12.69 %	12.59 %

I.R. 3300 (NH_2), 3410 (NH), 1355, 1150 (S = O) Cm^{-1}

(4) 7-Chloro-4-(-4'-Amino-2'-Methyl phenyl sulfonimido) quinoline.

Recrystallised from ethyl alcohol.

Melting point 265° Mol. formula $C_{16}H_{14}N_3O_2ClS$.

Yield 55 %

		Practical	Theoretical
Elemental Analysis	C	53.24 %	53.25 %
	H	4.012 %	4.020 %
	N	12.08 %	12.09 %

I.R. 3375, 3440 (NH) 1450, 1375 (S=O) Cm^{-1}

(5) 7-Chloro-4-(4'-Acetamido-phenyl sulfonimido) quinoline

Recrystallised from ethanol.

M.P. = 220° Molecular formula - $C_{17}H_{14}O_3N_3ClS$

Yield = 56 %

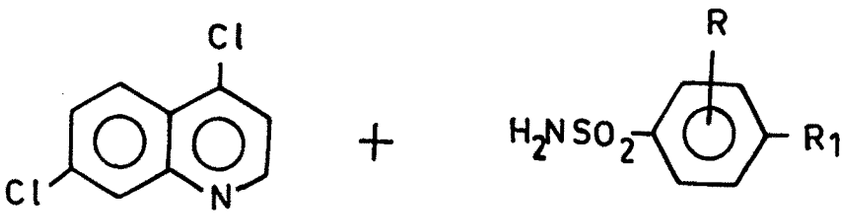
		Practical	Theoretical
Elemental Analysis	C	54.30 %	54.32 %
	H	3.71 %	3.72 %
	N	11.16 %	11.18 %

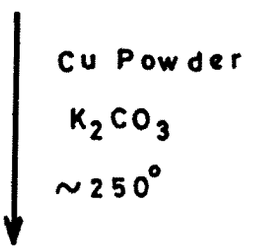
I.R. 3395, 3400 (NH) 1445, 1360, 1133 (S=O) Cm^{-1}

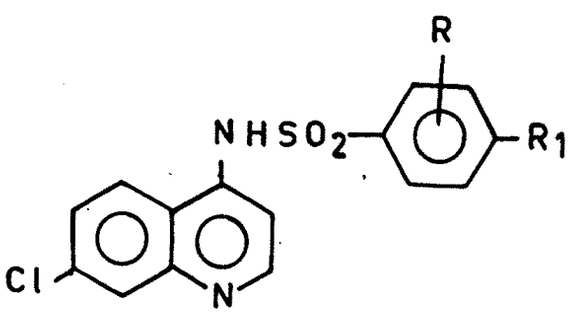
SCHEME-V

Synthesis of

N [7-chloro-4-quinolyl] sulfonamides







Compounds :–

	R	R_1
V	H	$NH-\overset{\overset{O}{\|\|}}{C}-CH_3$
VI	CH_3 meta to NH_2	$NH-\overset{\overset{O}{\|\|}}{C}-CH_3$
VII	H	CH_3

(6) 7-Chloro-4 (4' - Acetamido-3' Methyl Phenyl Sulfonimido) quinoline

Recrystallised from ethanol.

M.P. = 310° Molecular formula - $C_{18}H_{16}O_3N_3CLS$

Yield = 56%

		Practical	Theoretical
Elemental Analysis	C	55.44%	55.45%
	H	4.09%	4.10%
	N	10.77%	10.78%

I.R. 3340, 3440 (NH, NH_2) 1360 (S=O) Cm^{-1}

(7) 7-Chloro-4-(4'-Methyl Phenyl Sulfonimido)Quinoline

Recrystallised from ethanol.

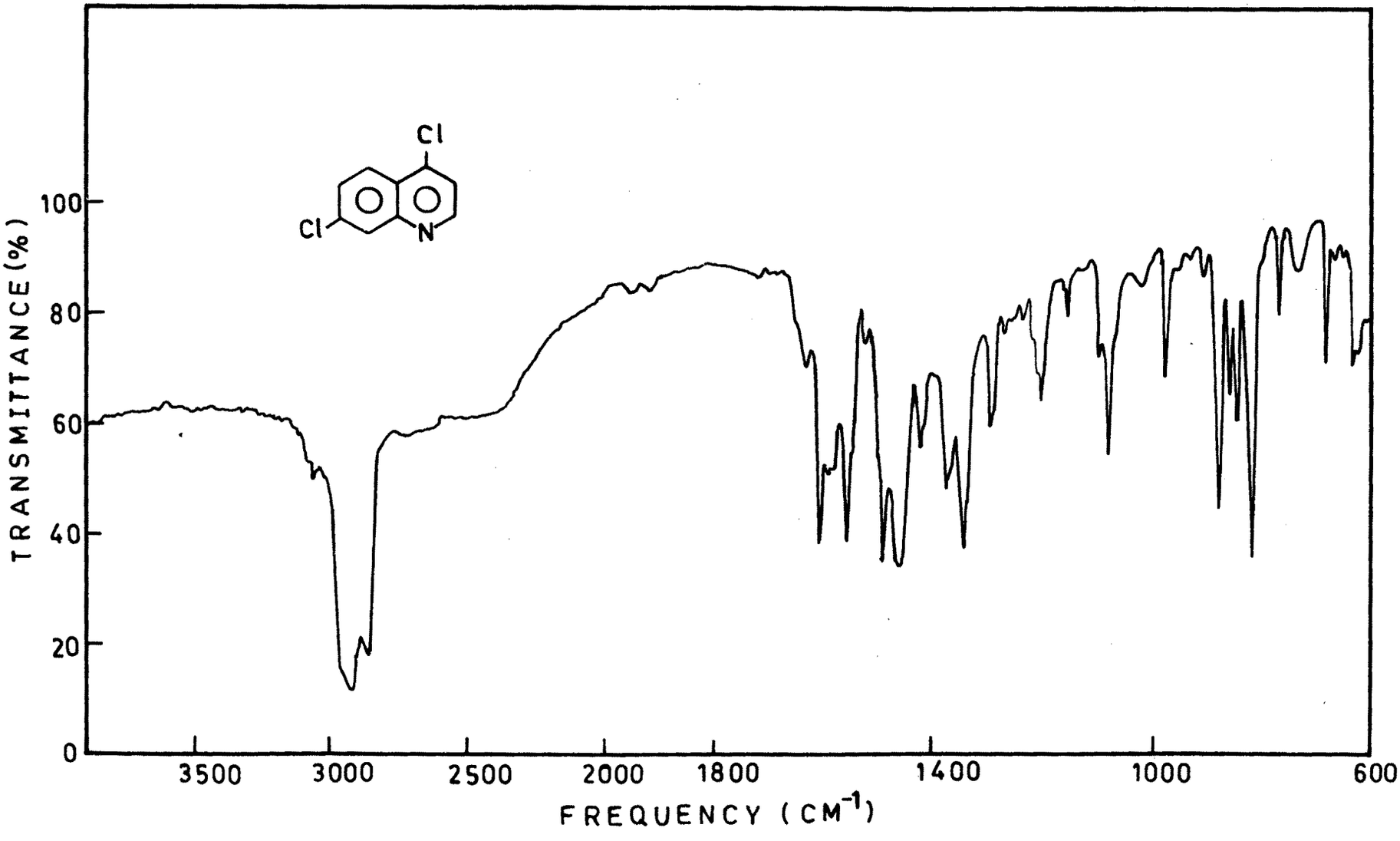
M.P. = 255° Molecular formula $C_{16}H_{13}O_2N_2CLS$

Yield = 50 %

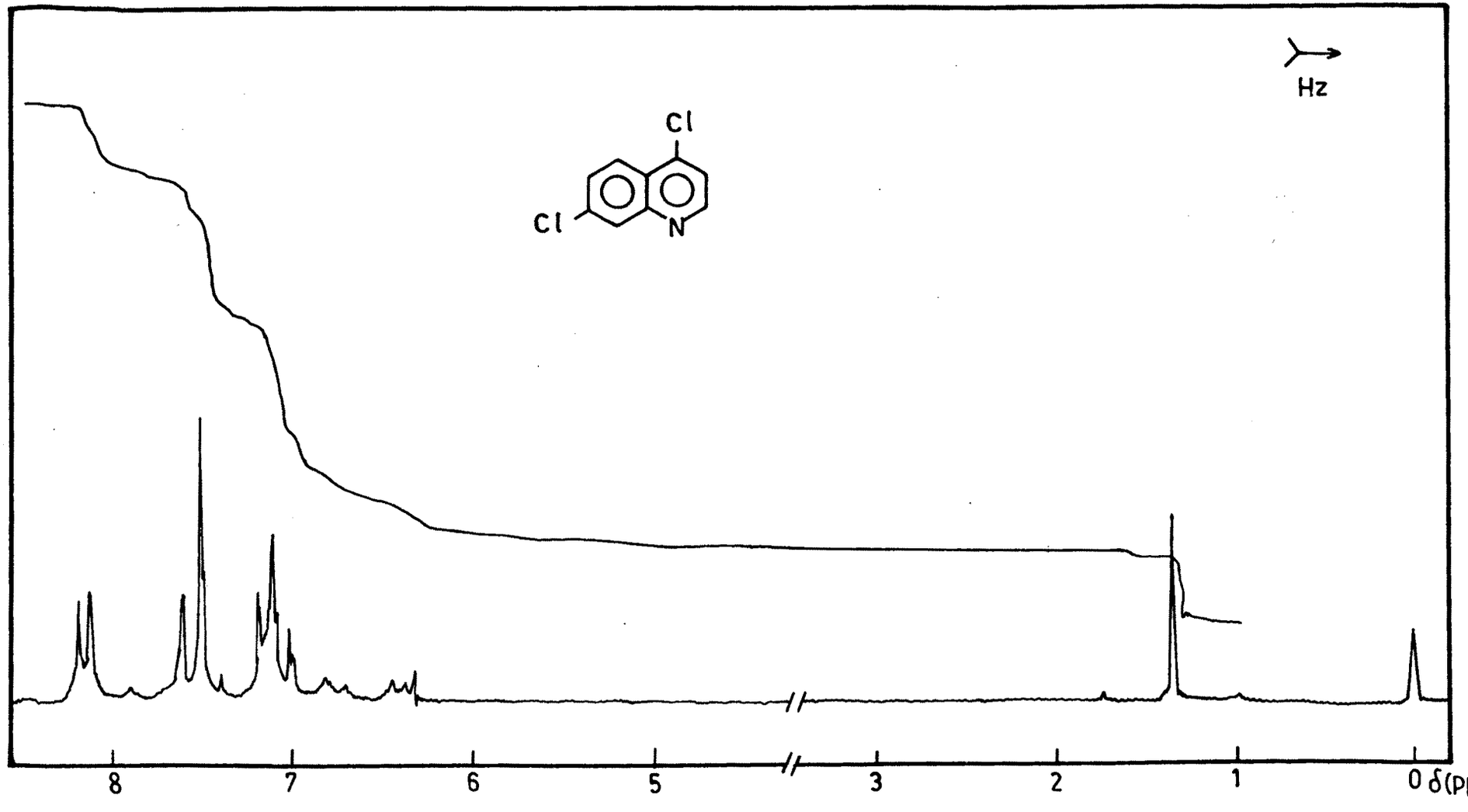
		Practical	Theoretical
Elemental Analysis	C	57.73%	57.74%
	H	3.89%	3.90%
	N	8.40%	8.42%

I.R. 3460, 3380 (NH) 1440, 1330, 1135 (S=O) Cm^{-1}

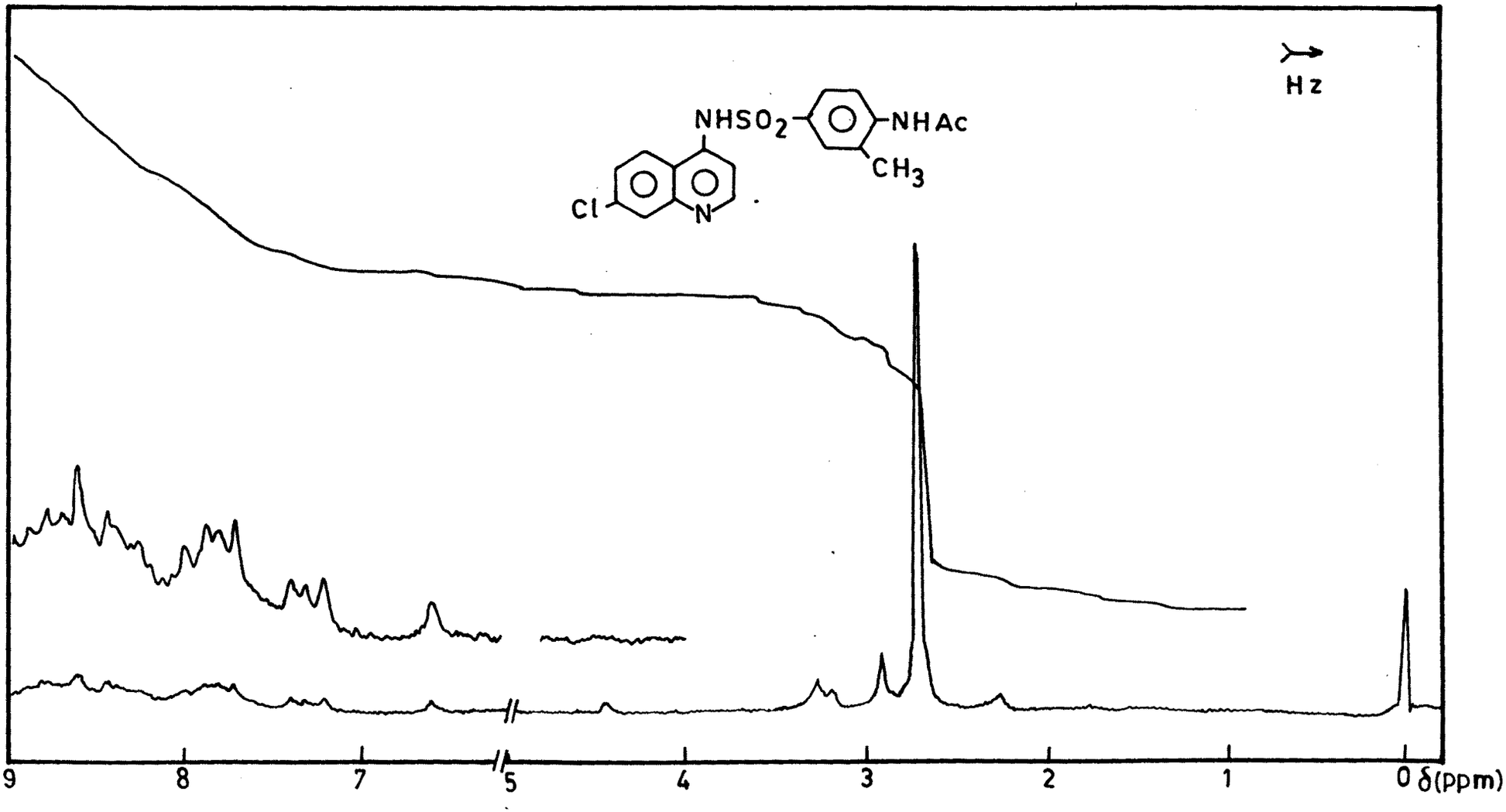
I R SPECTRUM OF 4,7-DICHLORO QUINOLINE.



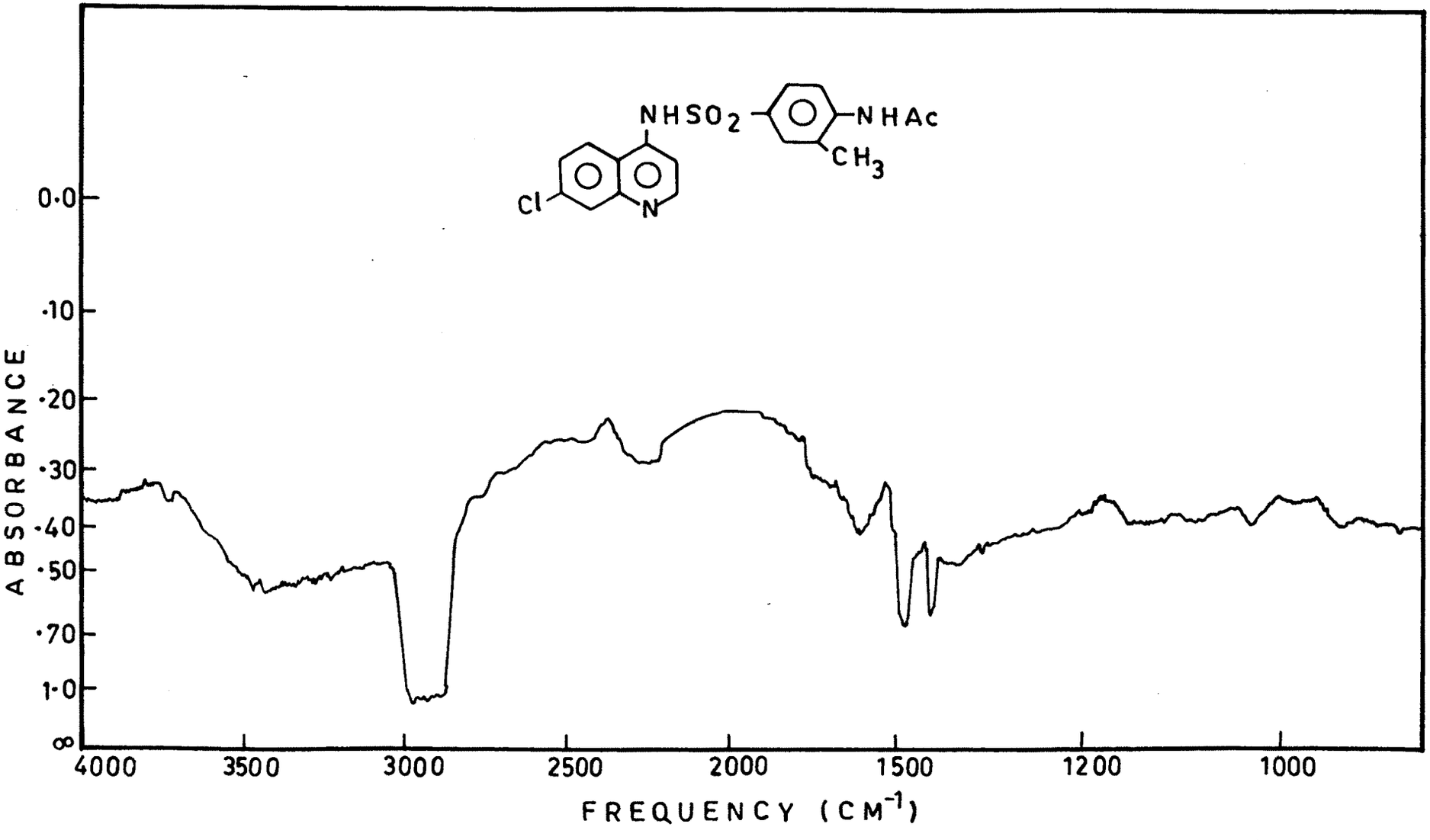
PMR SPECTRUM OF 4,7-DICHLORO QUINOLINE .



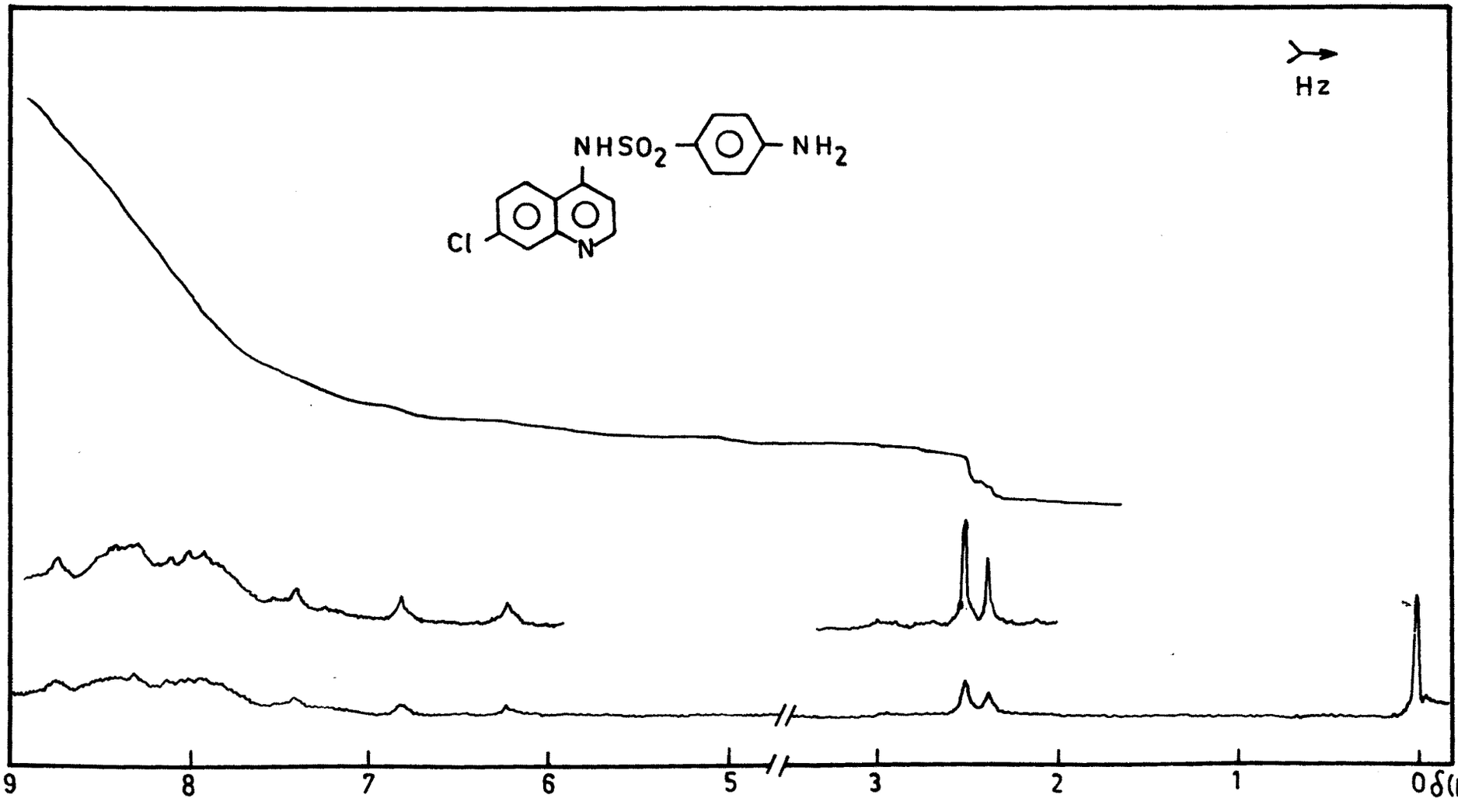
PMR SPECTRUM OF 7-CHLORO-4-[4'-ACETAMIDO-3'-METHYL PHENYL SULFONIMIDO] QUINOLINE.



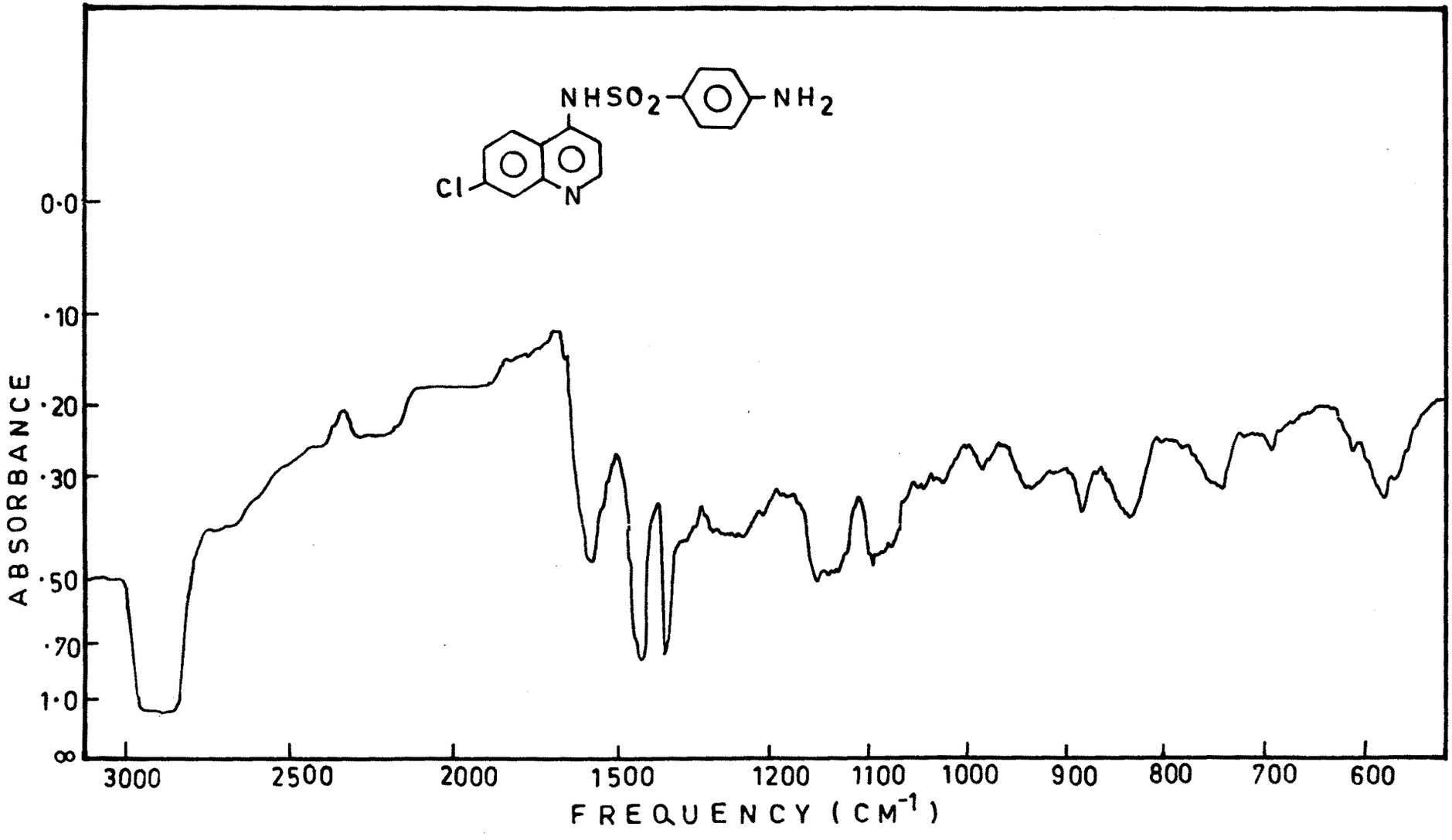
I R SPECTRUM OF 7-CHLORO-4-[4'-ACETAMIDO-3'-METHYL PHENYL SULFONAMIDO] QUINOLINE.



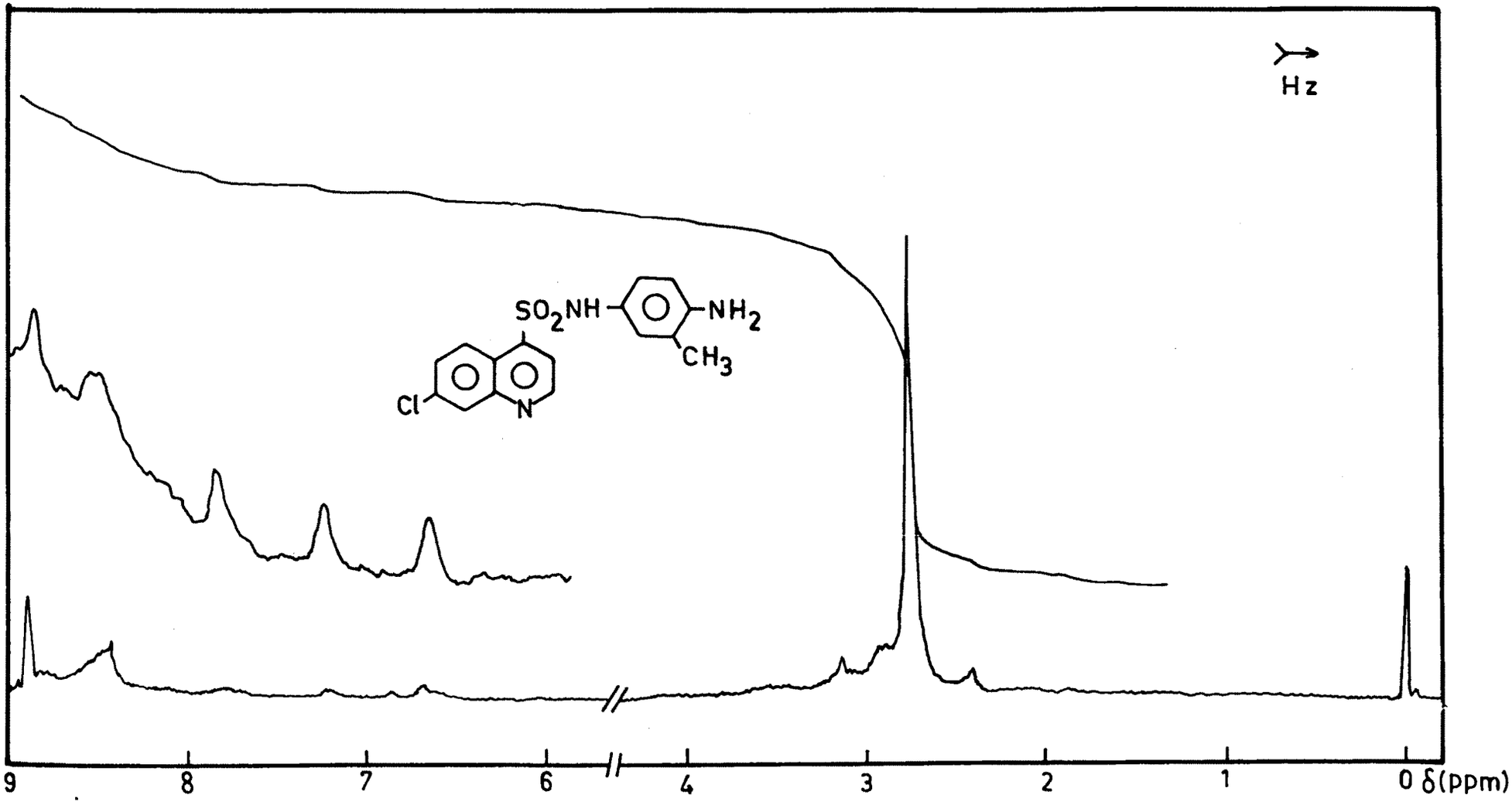
PMR SPECTRUM OF 7-CHLORO-4-[4'-AMINO PHENYL SULFONIMIDO]QUINOLINE .



I R SPECTRUM OF 7-CHLORO-4-[4'-AMINO PHENYL SULFONAMIDE] QUINOLINE.



P M R SPECTRUM OF 7-CHLORO-4-[4'-AMINO-3-METHYL PHENYL SULFONIMIDO] QUINOLINE .



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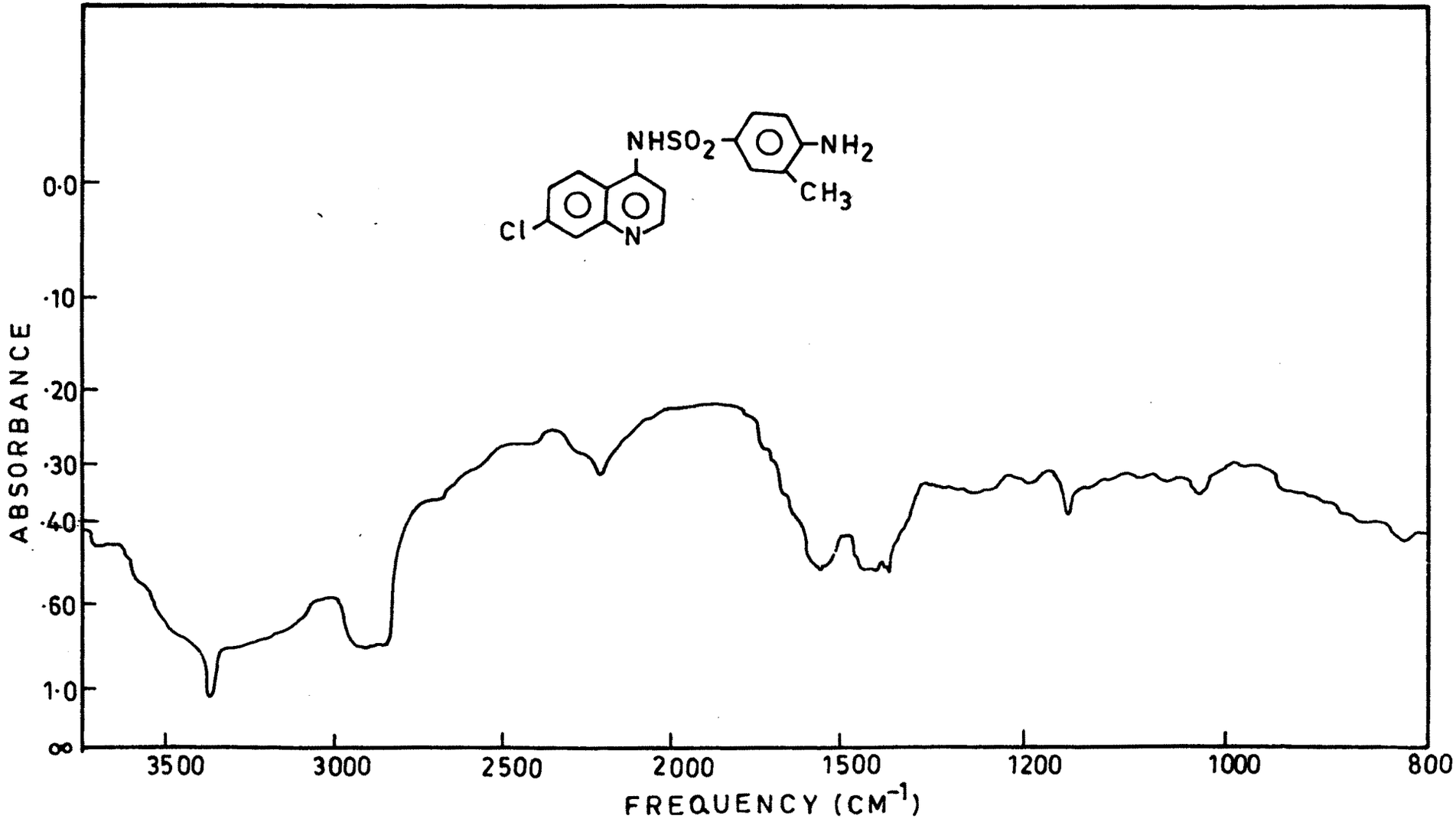

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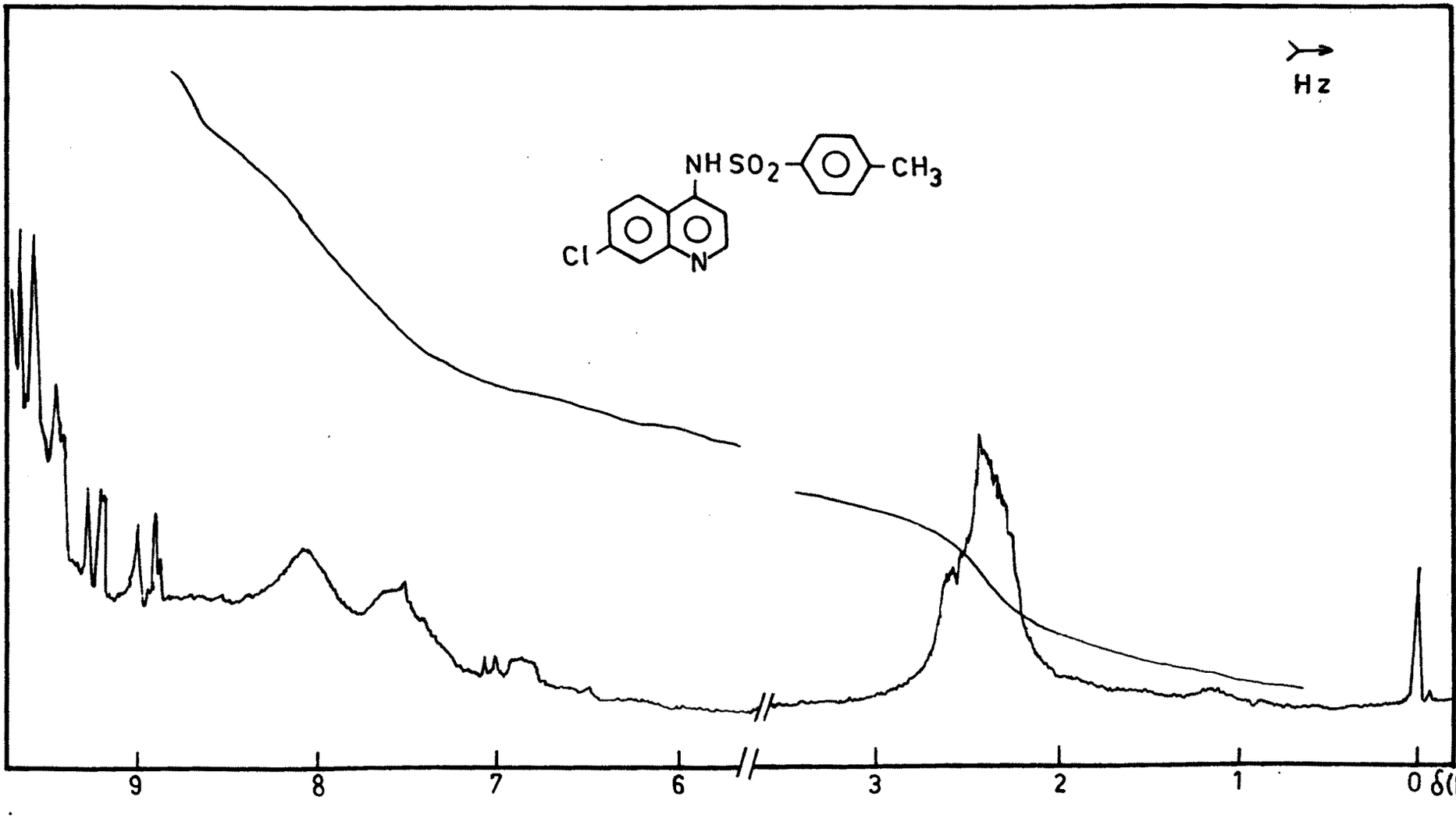

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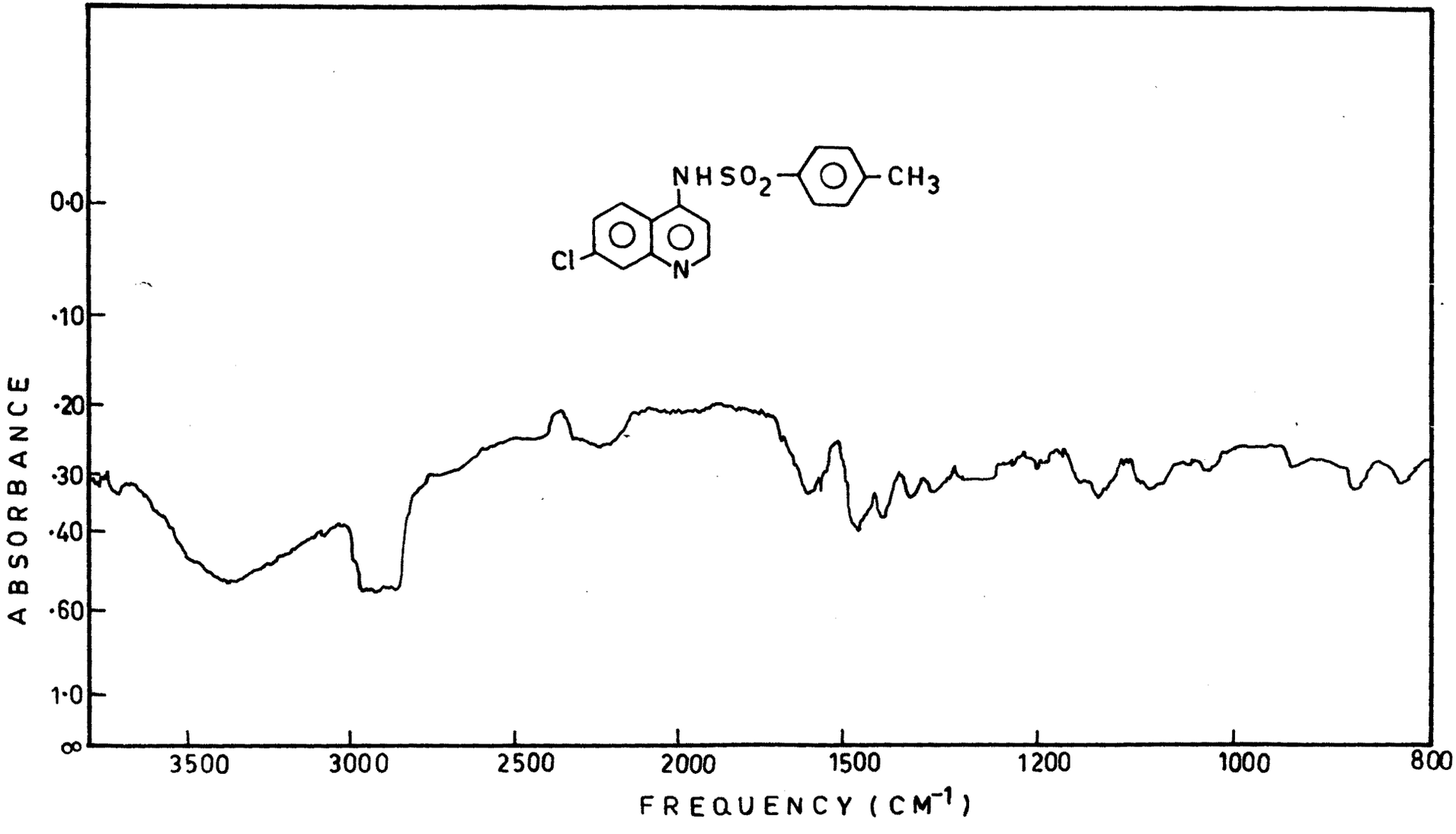

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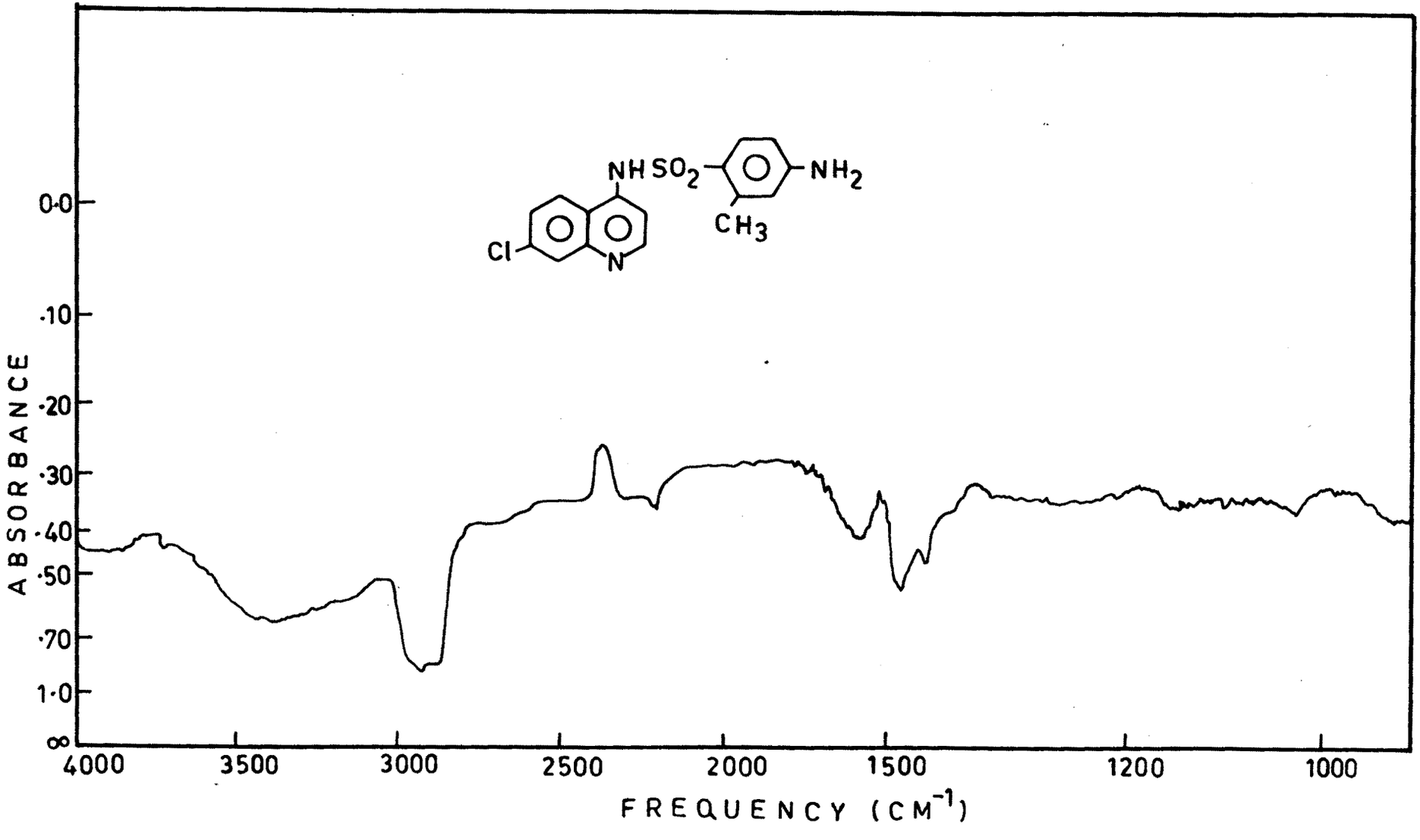

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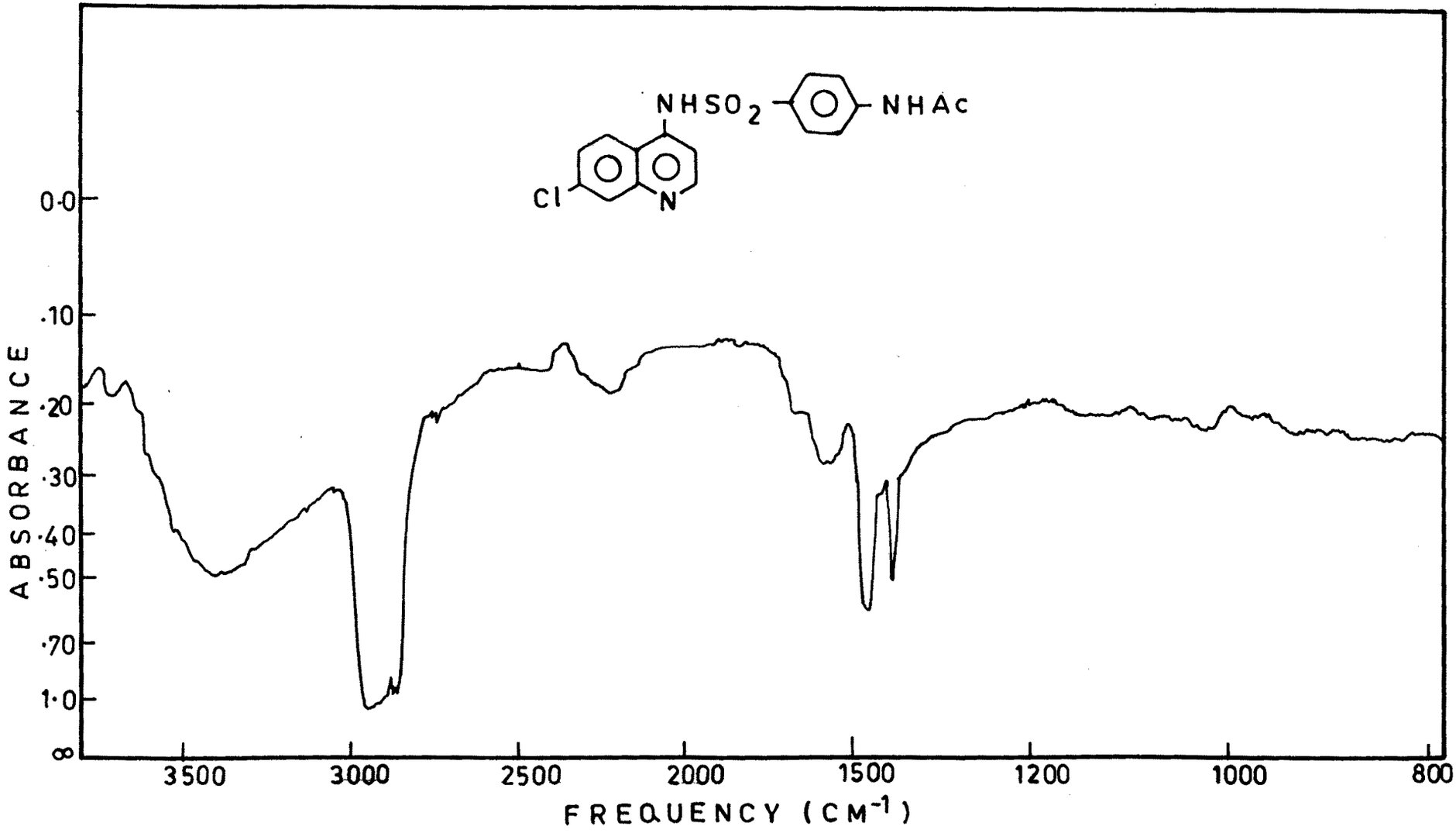

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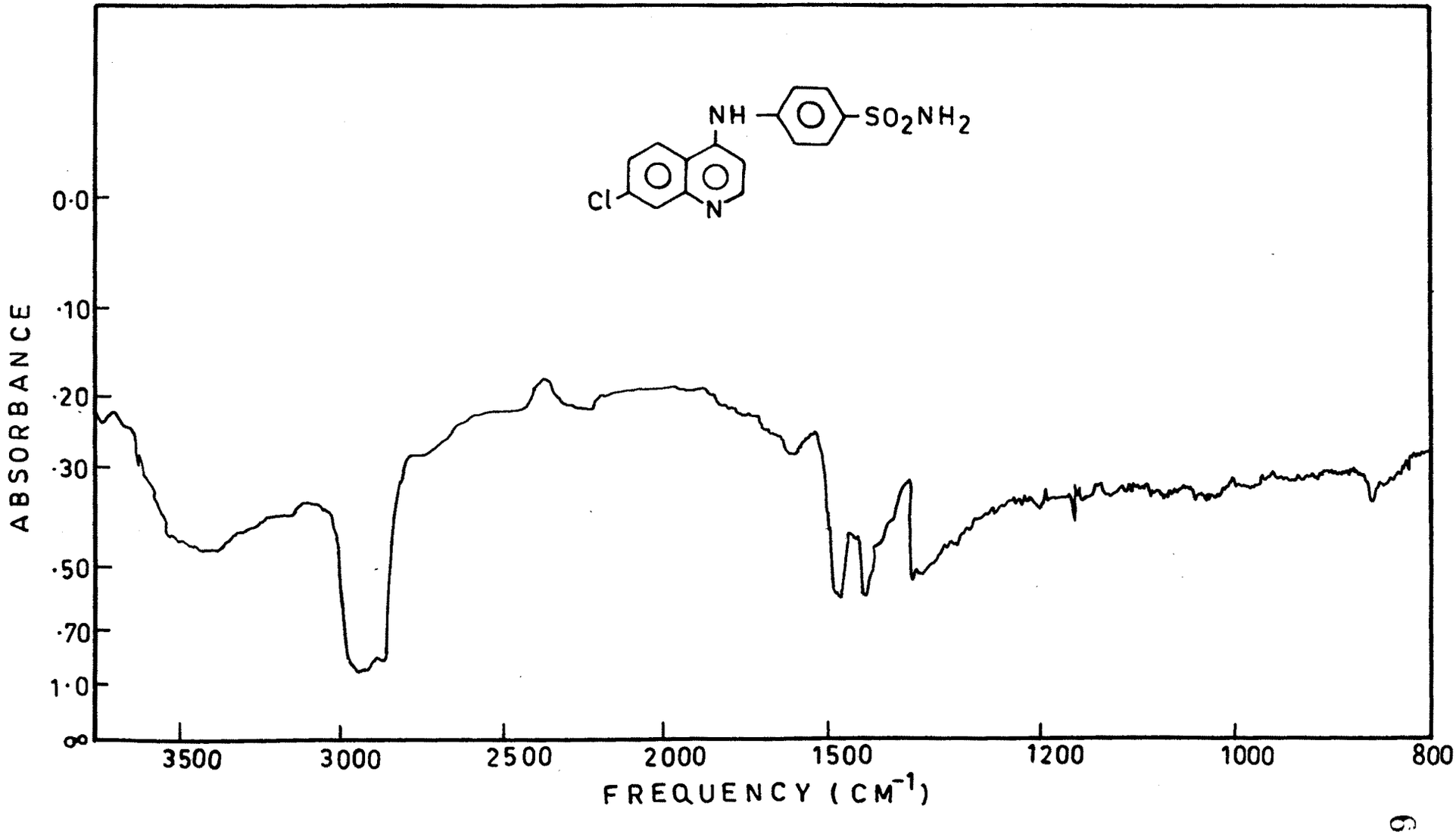

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