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Preparation, Characterization and Biological Activity of Some Novel Schiff Bases

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Abstract

The [C-1] was synthesized by reaction of acide red -37 with 3,4,5 tribromobenzaldehyd and [c-2] was synthesized by reaction of sulfamethaxazole with 3,4,5 tribromobenzaldehyd , the compounds [c-1] and [c-2] were characterized by elemental analysis , infrared ,¹H-NMR and mass spectroscopy .The biological activity were compared with amoxicillin as standard .

Keywords: 6-(3,4,5tribromobenzylideneamino)-5-(4-(3-(3,4,5tri bromophenyl)acrylamido)-2-Sulphatophenyl) diazenyl)-4-hydroxynaphthalene-2-sulphonicacid[c-1],4-(2-chloro3,4,5 trimethoxybenzylidene)amino-N-(5-methyl-isoxazol-3yl)-benzene sulfonamide [c-2]

Introduction

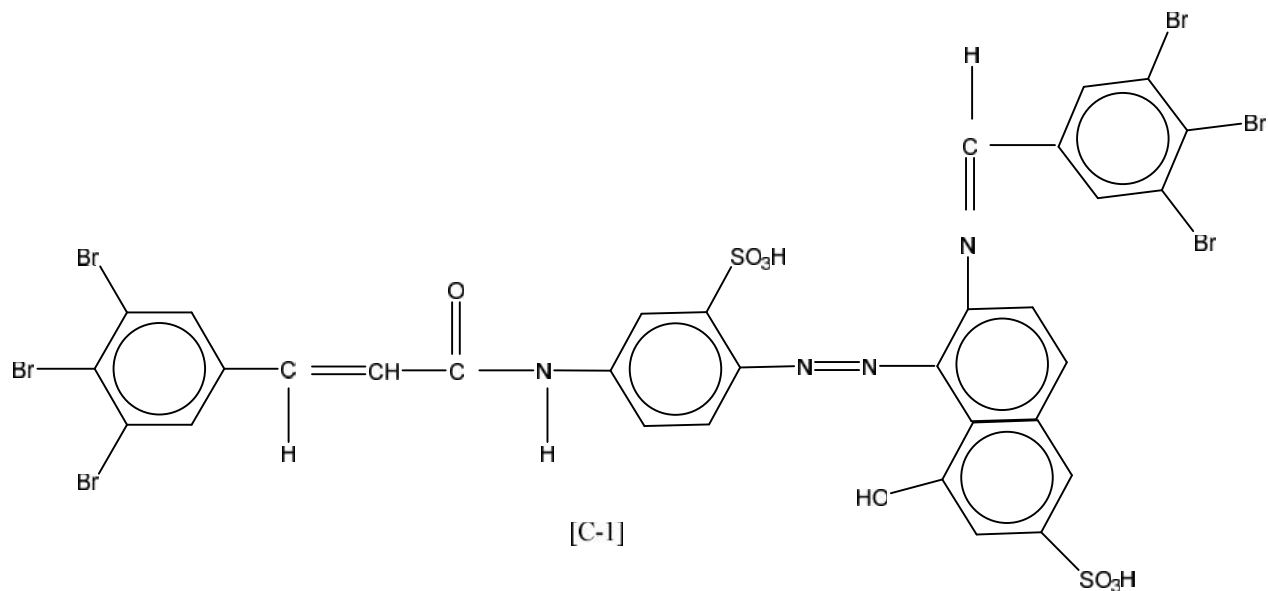
Schiff bases are an important class of organic compounds¹. They were reported by hugo Schiff in 1864². Schiff bases are widely studied and used in the fields of organic synthesis and metal ion complex^{3,4} for a number of reasons their physiological and pharmacological activity's^{5,6,7}.

Schiff bases are important compounds owing to their wide range of industrial applications⁸. Schiff's bases have also been shown to exhibit a broad range of biological activities ,including antifungal, antibacterial, antimalarial, anti-proliferative, anti-inflammatory, antiviral & antipyretic perperties.^{9,10}

Experimental

Instrumentation

Melting points were measured on Gallenkamp Electronic melting points apparatus, the elemental analysis was performed on a Perkin-Elmer 2400. Infrared spectra were recorded using potassium bromide disks on a Pye Unicam SP3-300 infrared spectrophotometer. ¹H-NMR experiments were run at 300 MHz on a Varian Mercury VX-300 NMR spectrometer using TMS as internal standard in deuterated dimethyl sulphoxide. The mass spectra were recorded on Shimadzu GCMS-Q-P 1000EX mass spectrometer at 70 eV.



Synthesis 4 (2-chloro-3,4,5-trimethoxybenzylidene) amino-N-(5-methyl-isoxazol-3-yl)-benzene sulfonamide (C-2)

A mixture of Sulfamethoxazole (0.01 mole) and 2-chloro-3,4,5-trimethoxybenzaldehyde (0.01 mole)

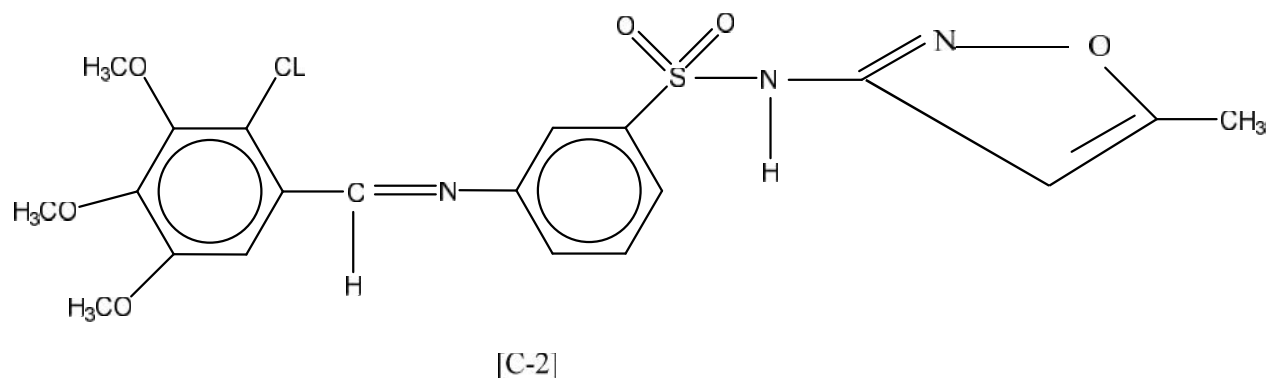
Synthesis of 6-(3,4,5-tribromobenzylideneamino)-5-(4-(3-(3,4,5-tribromophenyl)acrylamido)-2-sulphatophenyl)diazenyl-4-hydroxynaphthalene-2-sulphonic acid [C-1]:

A mixture of (0.01 mole) of acid red-37, (0.01 mole) of 3,4,5-tribromobenzaldehyde were mixed together in 200 ml dry ethanol. The mixture was heated to reflux for 12 hours at which a brown residue was separated.

The reaction mixture was then cooled and the light brown residue was separated by filtration. The solid was recrystallized from ethyl alcohol to give faint brown crystals.

and in a round bottom glass (Pyrex) flask (250 ml) in pure ethanol (100 ml) are stirred for 30 minutes.

The mixture was heated to reflux for 8 hours and kept overnight. The solid product was separated by filtration. The solid was recrystallized from ethanol.



Results and Discussion

Spectroscopic studies of 6(3,4,5tribromobenzylideneamino) -5-(4-(3-(3,4,5tribromophenyl)acrylamido) -2- sulphato phenyl) diazenyl) -4- hydroxynaphthalene -2 sulphonic acid [c-1].

The IR spectra of [c-1] table (1) exhibited a strong stretching frequency band for carbonyl group at 1671cm^{-1} and two absorption bands at 3312cm^{-1} due to (N-H). The $^1\text{H-NMR}$ spectrum of [C-1] in deuterated DMSO showed a singlet signal at 9.53 ppm due to proton of the NH group, singlet signal at 8.65 ppm suggested the attribution of the proton of the CH=N group, as well as multiplets in the rang 6.90-7.88 ppm due to the phenyl protons and the $^1\text{HNMR}$ spectrum exhibit doublet signal at 8.20 ppm due to (Ar -CH =CH, $J=12.3\text{Hz}$). The mass spectrum of [C-1] showed the molecular ion peak at m/z 1129.10(75%), the base peak at 65.17(100%).

Spectroscopic studies of 4(2- chloro 3,4,5 trimethoxy benzylidene) amino - N- (5- methyl - isoxazol - 3-yl)benzene sulfonamide {c-2}.

The infrared spectrum of [c-2] displayed tow absorption bands at 3246cm^{-1} due to N-H. $^1\text{HNMR}$ spectrum of the (c-2) in deuterated DMSO- d_6 .

Table 1 showed a singlet signal at 2.208 ppm suggested the attribution of the protons of the CH_3 group. The singlet signal at 6.110ppm suggeste the attribution of the proton of CH of the isoxazole ring, the multiplet signal at 6.715 – 7.782 ppm suggested the attribution of the protons of two aromatic benzene rings, the singlet signal at 8.825ppm due to proton of the CH=N group, and the singlet signal at 9.108 ppm due to NH group. The mass spectrum of [c-2] showed the molecular ion peak at m/z 465.21(89%), the base peak at 77.33(100%).

Biological activity:

Measurement of antimicrobial activity using diffusion disc method:

A filter paper sterilized disc (diameter 80mm) saturated with measured quantity of the sample is placed on plate containing solid bacterial medium (nutrient agar broth) or fungal medium (Dox's medium) which has been heavily seeded with the spore suspension of the tested organisms after incubation, the clear zone of inhibition surrounding the sample is taken as a measure of inhibitory power of the sample^{11,12,13,14..}

The compounds [c-1] and [c-2] were screened for their antibacterial activity against one gram positive bacteria, one gram negative bacteria and fungi candida albicans, the results of antimicrobial studies are given in table 3

Table 1 : spectroscopic for {c-1} and {c-2}

Compound number	IR (KBr) CM^{-1}	$^1\text{HNMR}\delta$ (PPM)
C-1	$\nu_{\text{C=O}}$ 1671 $\nu_{\text{N-H}}$ 3312	9.53(S,NH) 8.65(S,CH=N) 8.20(d, CH=CH) 6.90-7.88(m.Ar)
C-2	$\nu_{\text{N-H}}$ 3246	9.10(S-NH) 8.82(S,CH=N) 6.71-7.78(m.Ar) 6.11(S,CH:isoxazol ring) 2.20(S,CH ₃)

Table 2 physical data of the prepared compounds

Compound	MP.C/colour	Solvent yield %	MF (Mwt)	Elemental analysis Calcd/found			
				C%	M%	N%	S%
c-1	254-256 Brown	Ethanol 78	$\text{C}_{32}\text{N}_4\text{S}_2\text{O}_8\text{Br}_6\text{H}_{17}$ 1129.615	34.03	1.51	4.95	5.67
				33.97	1.09	4.28	5.23
c-2	162-164 yellow	Ethanol 90	$\text{C}_{20}\text{H}_{20}\text{N}_3\text{SO}_6\text{CL}$ 465.911	51.57	4.32	9.01	6.87
				51.10	3.96	8.88	6.13

Table 3 inhibition zones (mm) of compound [c-1] and [c-2]. The activity of 2,5mg/ml of sample Amoxicilline was used standard

Compound standard	<i>Staphylococcus aureus</i>	<i>Escherichia coli</i>	<i>Candida albicans</i>
c-1	14	26	13
c-2	18	20	17
Amoxicilline	23	21	15

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