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Synthesis, spectroscopic characterization of schiff bases derived from 4,4'-methylen di aniline

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ABSTRACT

The synthesis , characterization of three Schiff bases derived by condensation of 4,4`- methylen di aniline with different aldehydes such as Benzaldehyde, p-methoxy Benzaldehyde and p-chloro Benzaldehyde, P-methoxy Benzaldehyde and p-Chloro Benzaldehyde are reported. Spectroscopic techniques , including IR , ^{1}HNMR were used to identify the products.

Key words: 4,4`- methylen di aniline compounds; Schiff bases.

INTRODUCTION

3, 3`-Di amino-di phenyl–methan(DDM);Known as 4,4`-methylen di aniline (MDA), is used as inter mediate in the manufactour of poly urethane fums. It is anti oxidant for lubricating oils, rubber processing and preparation of azo-dyes.[1,2] MDA is a hazardous Substance that causes liver damages, skin and eye irritation[1-3] .On other hand, it was reported that some Schiff bases of aniline derivatives display anti inflammatatory and anti pyretic properties[4] . the general preparation of Schiff bases of metal complexes of these types of ligands were first-published in the 1860[5] . Schiff bases or imines have the general formula RN=CR` where there the R and R` are alkyle, aryl , cyclo alkyl or hetro cyclic groups. They are formed by condensation reaction that occurs when aldehydes and some ketones react with primary amines. Imines play animportent role in many biochemical reactions because some of the enzymes use an amine group of an amino acid to react with an aldehyde or ketone to form animine linkage. It follows then , that aldehyde or ketone react with diamines in astoichiometry of 2:1 , adiimine compound is produced[6] . In the present work , Schiff's bases of banzaldehyde, anisaldehyde and P-cl-benzaldehyde with 3,3`-diamino-diphenyl methan (S1 ,S2 ,S3) were prepared[7-11] and characterized .

MATERIALS AND METHODS

2 . 1. Materials and physical measurements :

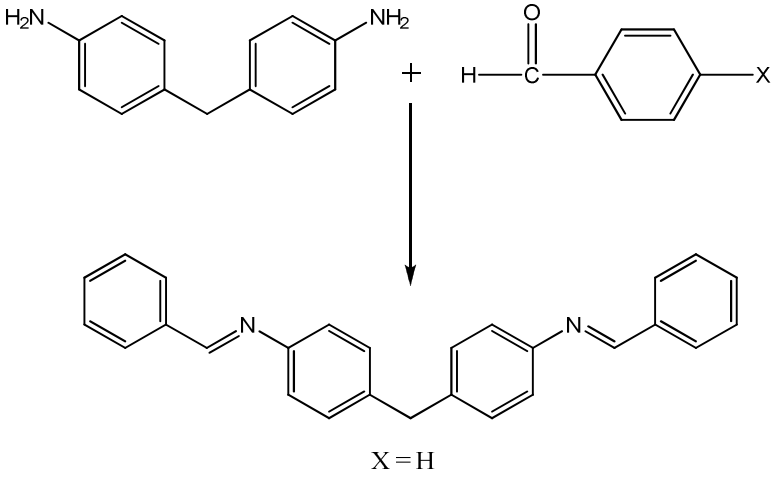
Benzaldehyde, p-chlorobenzaldehyde and p-methoxy benzaldehyde (Anisaldehyde) were purchased from (Fluka). The solvent grade and distilled prior to use. Melting point were determined on a BUCHI melting point 501,IR spectra were measured on shimiadzu spectrophotometer as KBr pellets in the region 4000 – 400 cm^{-1}, electron spectra were recorded by PG $T80^{+}$ Instrument. Central laboratory of Asfahan University(Iran). The ^{1}H–NMR spectra were recorded in DMSO-d^{6} on Bruker 400 MHZ spectrometer using TMS an internal standered in Iran.

2 . 2 . Synthesis of the Ligands (S1, S2, S3) :

Schiff's bases were prepared by addition of amine (8mmole) and ethanol (25 ml) in 100 ml conical flask equipped with a stirrer bar . stirring at room temperature about 20 min or until all the solid has dissolved . Add a solution of aldehyde (15 mmole) in ethanol (25 ml) and continue to stirrer the solution turns an intense golder yellow colour before the product precipitates over 30 min as a golden yellow powder . the product isolated in a quantitative yield

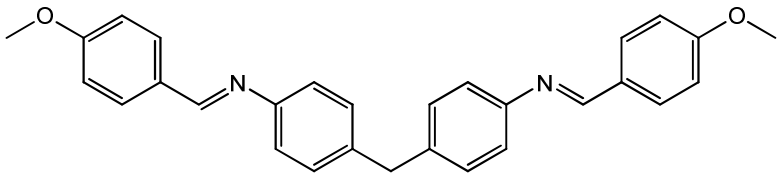
by filtration and washed with small quantities of ethanol to remove any un reacted starting materials. The product was recrystallized from a minimum volume of boiling ethanol .[12]

The structures of ligends are given in scheme (1) . The physical properties of these compounds (S1,S2,S3) are listed in table (1) .



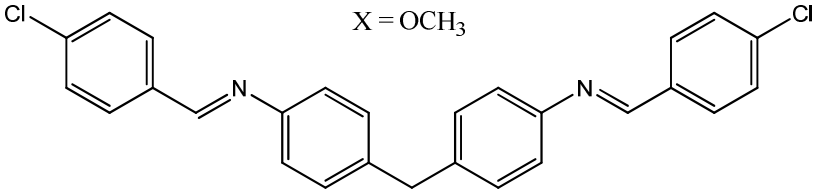
X = H

4.4`methylen bis(N-benzylidene aniline) (S1)



4.4'methylen bis(N-(4-methoxy benzylidene)aniline) (S2)

X = OCH_3



4.4'methylen bis(N-(4-chloro benzylidene)aniline) (S3)

X = Cl

Scheme (1) - The chemical structure of Schiff bases

Table (1) - Some Physical Properties for synthesized Schiff bases

NO.	Yield (%)	Color	M.P. (^{0}C)
S1	90%	Golden Yellow	130 -132
S2	85%	Golden Yellow	161 -162
S3	83%	Golden Yellow	181 -183

RESULTS AND DISCUSSION

3 . 2 IR Spectra :

The IR spectral data for the ν compounds are summarized in table (2). the medium band at 3000.17 – 3049.25 cm^{-1} are assigned to the aromatic C-H stretching , and aliphatic ν C-H (-CH_2 groups) at 2902.67 – 2950 cm^{-1} (asym) and 2877.60 – 2879.72 cm^{-1} (sym) .

As a medium – weak bands . The ν(C=N) bands appear as a strong bands at 1604.66 – 1625.99 cm^{-1} [13].

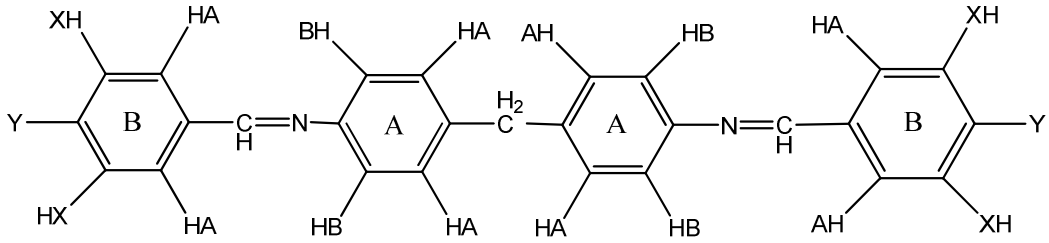
Table (2) : IR data for the compounds cm^{-1}

Compound	ν (C=C) aromatic	ν (C-H) aromatic	ν C-H (-CH_2) Aliphatic Asym.sym	ν (C=N)
S1	1450.47(s)	3030.17(w)	2950(m) 2879.72(m)	1625.99(s)
S2	1512.09(s)	3000.17(w)	2904.60(m) 2877.69(m)	1604.66(s)
S3	1589.23(s)	3049.25(w)	2902.67(m) 2879.52(m)	1623.65(s)

3 . 3 Proton NMR (^{1}H–NMR) Spectra :

^{1}H-NMR spectra of the investigated compounds which were dissolved in deuterated di methyl sulphoxide (DMSO-d^6) are shown in table 3.the solvent was showed two peaks , the first at 2.3 – 2.5 ppm due to DMSO solvent and the second at 3.8 ppm due to H_2O in DMSO [14,15] .

The structure of prepared compounds presented are in fig. (1)



Y = H (S1), OCH_3 (S2), Cl (S3).

Fig: The structure of compounds.

Ring's a protons showed AB system for all compounds at range 7.25 – 7.35 ppm because ΔV/J<10. Protons of ring B showed multiplet signal at 7.4 – 7.7 ppm in compound S1 while in compound S2 and compound S3 the first at 7.2ppm and the second at 7.8ppm . The signal of CH proton for C=NH group appeared at 8.65 – 8.52 ppm , while CH2 appeared as singlet signal at 4.1 – 4.3 ppm . CH_3 protons for OCH_3 group in compounds S2 showed a singlet peak at 3.1 ppm

Table (3) : ^{1}H-NMR for compounds.

NO.	Y	HC=N	Ring A(AB)	Ring B	CH_2
1	H	8.62 (s)	7.2 – 7.35	7.4 – 7.7 (m)	4.3 (s)
2	OCH_3	8.65 (s)	7.1 – 7.29	7.5 (d),7.9(d) J = 7.1	4.1 (s)
3	Cl	8.52 (s)	7.15 – 7.3	7.2 (d),7.89(d) J = 7.2	4.2 (s)

s : singlet
d : doublet
m : multiplet

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