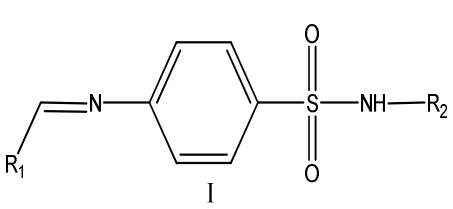
ABSTRACT

This study is concerned with the synthesis, characterization, biological activity and study of reduction – coupling of nitrone compounds with some ketones leading to electrosynthesis series of some new hydroxylamines.

Twenty three of Schiff base compounds I were synthesized bycondensation reaction of some sulfonamids with aldehydes.



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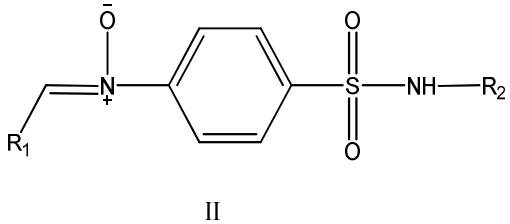

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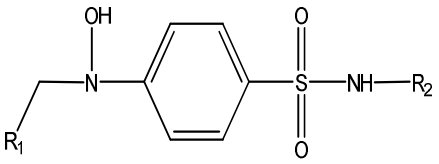

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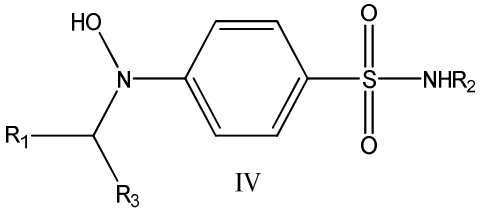

IV

R1=Ph ,o-OH-Ph,p-OH-Ph ,p-NO_2-Ph , C_3H_7

R_2=C_5H_6NO,C_3H_2NS,CH_3N_2,C_4H_4NO,$C_5H_5N_2$, C_5H_4N, $C_4H_3N_2$

R_3=$C_6H_{12}O$, $(CH_2CH_3)_2O$, C_8H_9O , C_8H_8BrO,$C_9H_{11}O_2$

A series of nitrones II have been synthesized by oxidation of Schiff bases by peracetic acid.

One of these nitrones was reduced by sodiumcyanoborohydride $NaBH_3CN$ to obtain hydroxyl amine III that can be used as a starting

material to prepare new nitrone on reacting it with other aldehyde or ketones.

Several nitrones were subjected by Cyclic Voltammetry (CV) in order to determine specifically the required potentials to reduction –coupling with numbers of ketones through electrosynthesis process. Thus, on adopting of CV measurements, some of synthesized nitrones subjected to electrochemical reduction-coupling with number of ketones to obtain IV.

The prepared compounds were characterized by using elemental analysis and F-TIR spectroscopy. Some of these are detected by UV-Visible, ^{1}H-NMR, ^{13}C-NMR and mass spectroscopy. The results are in good agreement with the calculated values and the results of other techniques.

UV-Visible spectra of nitrones, showed distinguished absorption bands within the regions (294-302 nm) in DMF and (290-304) in DMSO which may be attributed to the $\pi \rightarrow \pi *$ transitions of $C=N^{+}\text{-}O^{-}$.

The IR spectra of Schiff bases showed an absorption band in the range (1616-1662 cm^{-1}) which may be assigned to stretching vibrations of C=N. Also, IR spectra of nitrones showed two absorption bands in the ranges (1137-1186) and (1595-1650cm^{-1}) which are related to the stretching vibrations of N→O and C=N group, while chemical reduction for one of these nitrones to hydroxyl amine gives the IR spectrum which contains absorption for -OH band at region (3345-3500cm^{-1})and absence of absorption C=N group. This result emphasized the occurrence of reduction.

The antibacterial activity of synthesized nitrones compounds against Gram positive bacteria Staphylococcus aureus and Gram negative bacteria Escherichia coli showed that most synthesized nitrone compounds exhibited a good activity against bacteria, especially in

compounds S7 and N7 because of the presence of nitro group which is already a polar group one. The Schiff base S5 when converted to nitrone N5, the activity is changed from *E. coli* to *S. aureus* as a result of modification in polarity of compound, thereforeN5, showed very good activity as compared with standard drugs.

FTIR spectra of electro synthesized compounds provided similar results compared with chemical reduction. Also, the disappearance of C=N bands and, the appearance of absorption –OH group in vibration (3394-3498 cm^{-1}) have been noticed.

The ^{1}H-NMR spectra of nitrones showed singlet signal in the regions 7.94-9.73 ppm which is attributed to the proton of nitrone groups-CH=NO.

Also, the ^{1}H-NMR of aromatic protons of nitrones appeared at range (6.02-8.30 ppm). The ^{1}H-NMR spectra were shown in Figures (3-31) to (3-44). Also, some of nitrones characterized by mass spectra were showed the molecular ion M+ peaks.

Some selected nitrones and electrosynthesized compounds were studied by cyclic voltammetry and characterized by CHN analysis, ^{1}H-NMR, ^{13}C-NMR and mass spectra, which indicate that the synthesized compounds were as expected.