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СИНТЕЗ И ХАРАКТЕРИСТИКА СОЛИ СТЕНХАУС ИЗ ФУРФУРОЛА И АНИЛИНА

Аннотация: В этой статье соль Стенхауза была синтезирована в два этапа. Первый этап была реакция раскрытия фуруролового кольца с использованием перхлоратной кислоты, а второй этап реакция два моль анилина В соляной кислоте. . Состав выделен на основе инфракрасной спектроскопии ИК и ¹H-ЯМР-спектроскопии.

Ключевые слова: соль Стенхауса, анилин, фурурол, краситель, перхлорат, гетероциклические соединения.

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SYNTHESIS AND CHARACTERIZATION OF STENHOUSE SALT FROM FURFURAL AND ANILINE

***Abstract:** In this paper, Stenhouse salt was synthesized by two steps the first step was reaction of open the furfural ring using perchlorate acid and the second step with reaction 2 moles of aniline in HCl. The compound was characterized based on ¹H-NMR and infrared spectroscopy*

***Key words:** Stenhouse salt, aniline, furfural, dye, perchlorate, heterocyclic compounds.*

1.Introduction:

The Furfural is an important compound from naturel resources contains a heteroaromatic furan ring and an aldehyde functional group. It is used for multiple purposes, is an important compound in the chemical industry as it is widely used in the manufacture of medicines such "Nifuroxazide", cosmetics, perfumes, flavors and resins as well as in other products such as detergents and dyes. [1,2]

Our purpose is synthesize Stenhouse salt, which have an great importance in the dye industry Stenhouse salts are among the well-known organic salts such as Schiff's bases and they are produced from the interaction of Furfural with 2-mol of aromatic (primary - secondary) amines in the presence of acid ,where the reaction undergoes furan ring

opening and produces a colored crystalline salt having a conjugate double bond open chain structure, Schiff reported that tertiary amines such as dimethylaniline gave no formation of coloured salts under such conditions,[3,4,5]

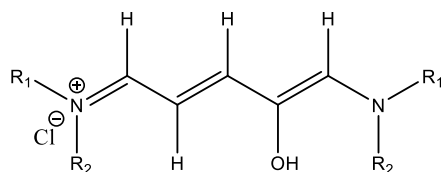


Figure 2. Structural formula of Stenhouse salts

There are many numbers of analytical applications to Stenhouse salts such as a reagent to detect furfural in oil, inhibitor for corrosion of steel and as a reagent in spectrometry and as dye for polyester fibers. [6]

In this paper the synthesis of Stenhouse salt have been reported form the furfural and aniline which it is possible to use a compound in the Synthesis of dyes

2- Experimental:

2.1. Materials:

Reagent grade 2-furfuraldehyde and aniline were used after purification. Perchloric acid (72%) was purchased from S.D. Fine Chemicals Ltd., Mumbai, India, and was used as received. All the solvents and chemicals were used of analytical reagent grade without further purification. Spectroscopic grade solvents (Merck-India, Ltd.) were used for UV–Visible, 1H-NMR spectroscopy and TLC plates were used also from Merck-India,

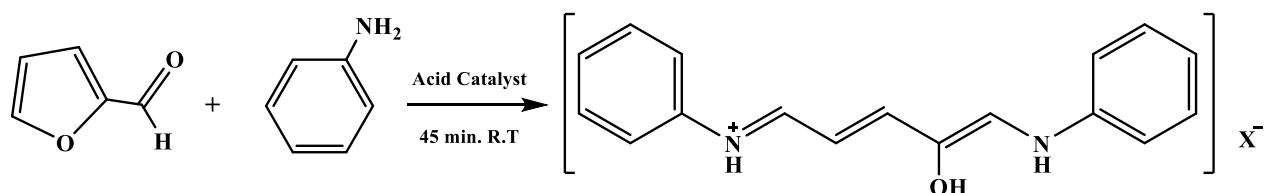
2.2. Methods and instruments

Melting points were determined by open capillary method, The Infrared spectra FT-IR were recorded in the range (400-4000 cm⁻¹) by KBr pellet from the Japanese company Jasco spectrophotometer. The Proton nuclear magnetic resonance 1H-NMR and the Carbon-13 nuclear magnetic resonance 13C-NMR device 400 MHz model by Switzerland company, thin layer chromatographic of aluminum coated by Silica Gel, UV–Visible absorption spectra were scanned on Shimadzu A-20 Spectrophotometer

using dye concentration of $1 \cdot 10^{-3}$ mg/ml in spectroscopic grade DMF. Buchner funnel to filter the product, melting points were determined by open capillary method.

2.3. General procedure for the synthesis of Stenhouse salts

Prepare Stenhouse Salt (SH¹) by adding equal amount of perchloric acid (72%) dropwise to the methanolic solution of aniline (0.01 mol) with continuous stirring at room temperature during 45 min. The resulting solution was cooled to 5 °C and 2-furfuraldehyde (0.01 mol) was added gradually with stirring, followed by the addition of a second portion of methanolic solution of aniline (0.01 mol) containing HCl as catalyst during of 30 min. The resultant reaction mixture was stirred for half an hour below 5 °C. The red product precipitated was filtered off, washed several times with water and dried in air giving red color Stenhouse salt (79%).



Scheme 1. General Scheme for synthesis of Stenhouse salt (SH¹)

Table 1. shows some physical properties and yield of Stenhouse salt (SH¹).

Stenhouse salt	Color	Melting point	Yield
SH ¹	Red powder	167-168 °C	79%

3. Results and discussion:

3.1. Synthesis and characterization of Stenhouse salt

((1Z,3E,5E)-1-(phenylamino)-5-(phenylimino)penta-1,3-dien-2-ol):

SH¹: ¹H-NMR (400 MHz, DMSO): d =4.90 (s, 1H, NH), 5.28 (s, 1H, OH), 5.51 (d, 1H), 5.74 (t, 1H), 6.36 (s, 1H), 6.77 (s, 1H), 6.94–9.03 (m,10H), 9.86 (s, 1H, +NH).

IR (KBr) cm⁻¹: m = 3202 (OH & NH), 1326 & 1228 (OH), 1110 (CO), 1555 (NH), 753 (NH), 2992 (conjugated alkene), 1623 (C,C of aromatic ring).

(Scheme 1) shows the synthesis of Stenhouse salt (SH¹) by the reaction 2-furfuraldehyde with two moles of aniline in the presence of mineral acid (HCl or HClO₄) to form crystalline red color products. this salt was obtained in good yields (79%).

The structure of these salt was established by spectral studies (IR, ¹H-NMR). The IR spectra of the salt exhibit the characteristic absorption bands at 1650-1600 cm⁻¹ (C,C conjugated), 1400–1350 cm⁻¹ (C,N) and 1350-1280 cm⁻¹ (aryl sec. –NH–). ¹H-NMR spectra of salt were in good agreement with the proposed structure (Scheme 1). In the ¹H-NMR spectra of Stenhouse salt (SH¹), the signal due to 1H of secondary –NH– Stenhouse salt appeared at d value of a 9-10 ppm was resulted from the reaction of aniline and furfural, and showed the absence of corresponding 2H signal of primary –NH₂ of aniline. Analogously, signals of 3H of furfural ring at 7.51, 6.67 and 6.49 d ppm were also absent indicating the ring opening reaction of furfural.

4. Conclusion:

In this paper, the Stenhouse salt (sh¹) was successfully synthesized with a yield of (79%), the salt structure was confirmed by the analytical spectra (NMR, IR) obtained. The resulting salt can be interacted with many diazonium salts derivatives and obtain pigments from It is expected to have important applications.

5. Acknowledgment:

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