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Synthesis of 2-Nitro-2- [4-(Phenyl imine) cyclohexa-2,5-dien-1-yl] Propane-1,3-diol

[1*]T. Bhaskar, [2]C.V. Mythili

[1*,2] Rani Anna Government College for Women, Tirunelveli-8

(Affiliated to Manonmaniam Sundaranar University, Tirunelveli-8)

Email: bhas51@yahoo.com

Abstract

Bronopol is an active organic compound against a broad spectrum of bacteria including gram-negative species that are resistant to many antibacterial agents. Bronopol is used in consumer products as an effective preservative agent as well as a wide variety of industrial applications. A novel synthesis of 2-Nitro-2-[4-(Phenyl imine)cyclohexa-2,5-dien-1-yl]Propane-1,3-diol from Bronopol (2-nitro-2bromo-1,3-propanediol) is carried out. Several attempts were made to synthesis bronopol derivatives of 2-Nitro-2-[4-(Phenyl imine) cyclohexa-2,5-dien-1-yl]Propane-1,3-diol from Bronopol the suitable methods were found . Bronopol reacts with Diphenylamine with suitable conditions gave the product 2-Nitro-2-[4-(Phenyl imine) cyclohexa-2,5-dien-1-yl]Propane-1,3-diol. The nature of the compound is a blue solid and the structure is confirmed by spectral studies.

Key words: Bronopol, diphenyl amine, ethyl acetate, 2-Nitro-2-[4-(Phenyl imine) cyclohexa-2,5-dien-1-yl]Propane-1,3-diol.

I. Introduction

Early work by Hodge, Dawkins and Kropp (1954) and by Zsolnal (1961) suggested that germinal bromonitroalkanes had antifungal activity. The broad-spectrum antibacterial properties of 2-bromo-2-nitro-1,3-diol have been described in a preliminary communication by Croshaw, Groves and Lessel (1964) and in comparison, with other members of a series of antimicrobial aliphatic halogen-nitro compounds by Clark et al (1974). Bronopol is used as a preservative in various cosmetic, toiletry and household preparations particularly because of its high activity against Gram-negative bacteria, especially *Pseudomonas aeruginosa* and other species. These organisms are common residents in water and as such can cause contamination and spoilage problems in cosmetics and toiletries (Morse et al, 1968, Skyes et al, 1969, Smart et al, 1972, Malcolm, 1975).

Pseudomonas are frequently implicated, particularly in oil-in-water emulsions which contain a significant number of non-ionic surfactants (Smart et al, 1972, Tenenbaum, 1971, Thomas et al, 1973). Bronopol is an effective antimicrobial preservative over a wide pH range. It is stable at acidic pH and is also useful as a liable antimicrobial preservative in alkaline media. Because of its broad-spectrum antibacterial activity, bronopol can also be used as an active agent, for example, in aerosol formulations. Bronopol has been reported to show persistent activity on the skin, as reported by Marples and Kilgman (1974); this contrasts with the fact that in vitro, it has been shown to have a weak growth-inhibitory effect on cultured human skin cells by Onoda and Saito (1974).

Bronopol is also called as 2-bromo-2-nitro propane 1,3 –diol. Bronopol is used as an antimicrobial preservative to protect spoilage due to microbial contamination (Kajimura et al, 2008). Bronopol is active against a broad spectrum of bacteria including gram-negative species. They are resistant to many antibacterial agents (British Pharmacopoeia, 1993, Wailhanser et al, 1983). It decomposes in alkaline solutions and at elevated temperatures and is acidic in nature.

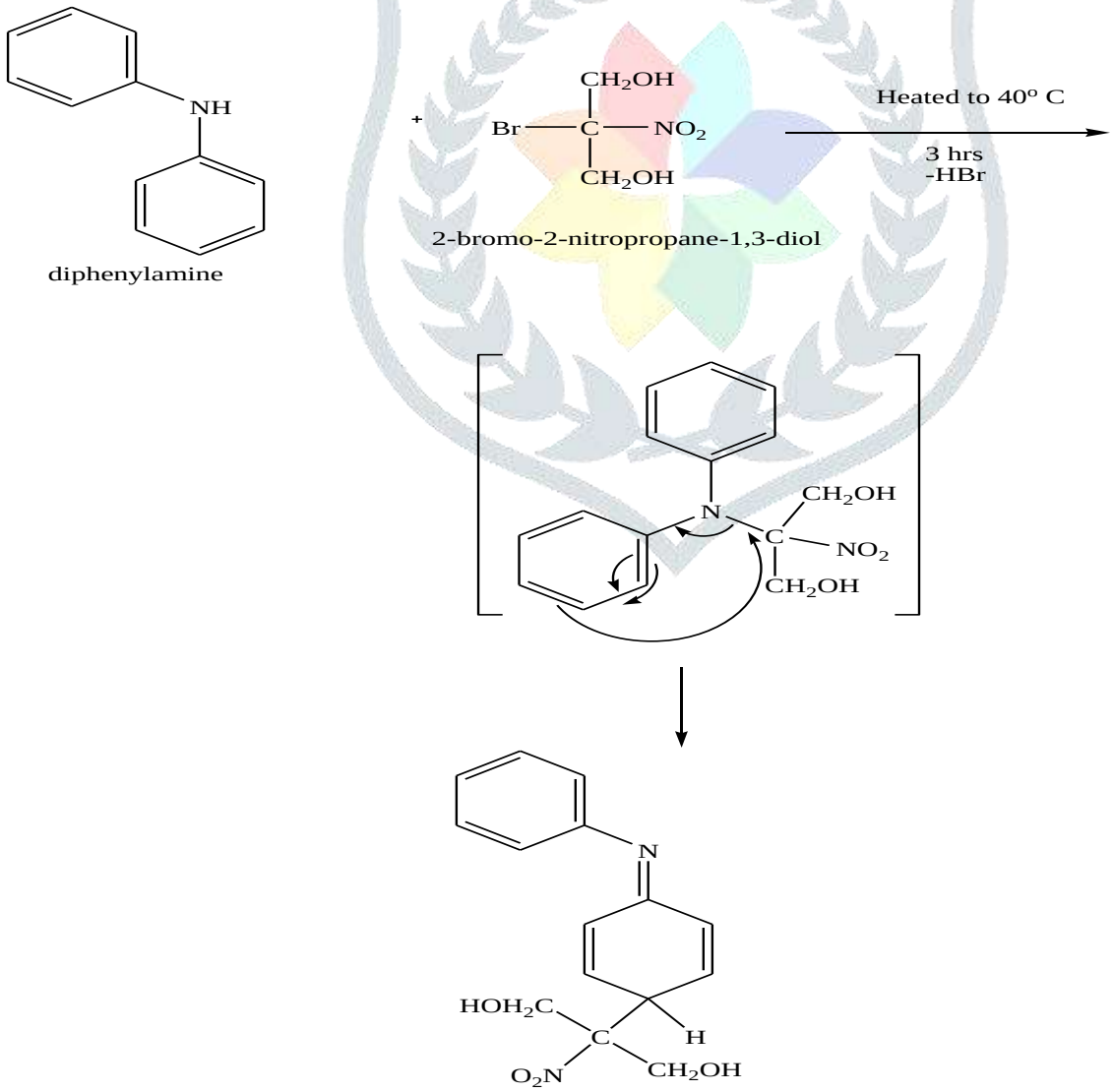
The color of the substance that has been obtained by the reaction of aliphatic 2-nitro-2-bromo propane-1,3-diol with diphenyl amine has been the subject of investigation by Wieland and Keharmann (Boyce et al, 1978, Chamberin, 2013). In the present study, 2-Nitro-2-[4-(Phenylimine) cyclohexa-2,5-dien-1-yl] Propane-1,3-diol has been synthesized and characterized by spectral studies.

II. Materials and Methods

Reagents and materials are AR Grade Diphenyl amine, Bronopol, Ethyl acetate. TLC 60F254 pre coated plates were used and the spots were rendered visible by exposing iodine vapour. FTIR-4700 spectra for IR spectra. NMR spectra were measured from Brucker, 400 MHz Narrow Bore FT-NMR Spectrum.

III. Experimental

About 0.1 mole of Bronopol was taken in a two necked round bottom flask and dissolved in ethyl acetate. About 0.1 mole of diphenylamine was dissolved in ethyl acetate taken in the equalizer funnel. Diphenylamine solution was added in drop wise manner so that the pH of the reaction mixture was maintained. After addition of the diphenylamine solution, the flask was heated to the temperature of about 60ºC. A blue-colored solid product was formed, which was filtered and dried. After 3 hours, TLC was run with help of a mixture of benzene and ethyl acetate.



Scheme

The structure of the compound was confirmed via spectroscopic studies. IR shows the following frequency: 3427 cm^{-1} OH stretching, 2932 cm^{-1} -CH aliphatic stretching, 1634cm^{-1} –C=C- stretching of alkane, 1354 cm^{-1} –NO2 stretching and 1022 cm^{-1} –O-C- symmetric stretching.

^{1}H-NMR shows at δ 6.92 to 7.26 aromatic proton, δ 5.64 alkene proton, δ 4.2 CH_2O proton, δ 2.166-OH proton, δ 1.66 alkane proton and $CdCl_3$ solvent impurity at δ 1.25. [5]

^{13}C-NMR shows -CN at 143 ppm, aromatic at 121-129 ppm, alkene at 117 ppm and CH_2O at 66.45 ppm and DEPT 135 shows CH_2O shows at 66.5 ppm.

IV. Results and Discussion

The reactions of the synthesized 2-nitro-2- [4-phenyl imino) cyclohexa-2,5-den-1-yl] propane-1,3-diol is given in the scheme. Due to the solubility of Bronopol in organic solvents and the reactivity with organic substances several trials were attempted. This reaction was carried out by using equalizer. Diphenyl amine reacts with Bronopol gives 2-nitro-2-[4-(phenylimino) cyclohexa-2,5-dien-1-yl] Propane-1,3-diol. The structure of the synthesized compound was established on the basis of spectral data.

V. Conclusion

In the present research work, bronopol derivative was synthesized from bronopol. The novel 2-nitro-2-[4-(phenyl imino) cyclohexa-2,5-dien-1-yl] Propane-1,3-diol was characterized by NMR, ^{13}C-NMR, DEPT135 and IR spectra.

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