



Research Paper

Synthesis of-3-Hydroxy-3-Propyl-1-p-Sulphonato (Sodium Salt) Phenyl Triazene.

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ABSTRACT

Hydroxytriazenes serve as useful group of chelating agents. These compounds have been named in three different ways.

(1) Hydroxytriazenes (2) Diazohydroxyamines (3) Triazene oxides.

The name hydroxytriazene has been used throughout in the present work. Four reviews¹⁻⁴ have been reported on the work done on the synthesis and analytical applications of hydroxytriazene

Key words: hydroxytriazene, Diazohydroxyamine Triazene oxides.

I. Introduction.

There are three methods known for the synthesis of hydroxytriazenes.

First Method:

This method involves reduction of nitrosobenzene or substituted nitrosobenzene with phenyl or substituted phenyl hydrazins to give corresponding hydroxytriazene. This method has been reported by Bamberger et al⁵, Gebhard and Thompson⁶ and Fischer et al⁷.

Second Method:

This method has been reported by Bamberger et al⁸⁻¹¹. It consists in coupling alkyl or aryl hydroxylamine with a diazonium salt in acetate buffered medium ($\text{pH} \cong 5$) at 0 to 5°C to give corresponding hydroxytriazene.

Third Method:

This method has been reported by Mitsuhashi and Simamara¹² consists in oxidation of diazoaminobenzenes with peroxy benzoic acid under mild conditions in a suitable solvent (ether, methanol and toluene) to give corresponding diaryl hydroxytriazene.

The second method gives better yields and is most widely used. Most of the hydroxytriazenes reported are prepared by this. In the present work also the second method has been used. method. for the preparation of hydroxytriazenes.

3-Hydroxy-3-propyl-1-p-sulphonato (sodium salt) phenyl triazene:

The synthesis of this compound consists of following stages:

Preparation of propyl hydroxylamine :

(Note: $\frac{M}{5}$ sulphamic acid has been taken in the preparation correspondingly (17.8 gm) nitropropane should have been taken for reduction purpose. It was observed that taking 25% more of nitropropane for reduction improved the yield of the final product. As such 22.25 gm of nitropropane was taken for reduction.

The propyl hydroxylamine has been prepared by the reduction of nitropropane (22.25 gm) in 300 ml water with ammonium chloride (30 gm) and zinc dust. Zinc dust (30 g) was added to the mixture in small bits with mechanical stirring in such a manner that the temperature of the reaction mixture was maintained between 0° to

15°C. The mixture was further stirred for 15 minutes and filtered under suction and washed with ice cold water. The filtrate was kept in an ice bath at 0° to 15°C. This solution of propyl hydroxylamine was used as such.

Diazotisation:-

Sulphanilic acid (34.6 gm) was dissolved in solution of sodium carbonate (10.6 gm in 100 ml) and cooled to 5°C. Sodium nitrite (13.8 gm) was dissolved in minimum quantity of water. This solution was added to the sulphanillic acid at 0° – 5°C in small lots at regular intervals.. In a 500 ml beaker conc. HCl (80 ml) was taken. The beaker was placed in a freezing mixture.

Under constant mechanical stirring the above solution of sulphanilic acid was added in small lots at regular intervals to the conc. HCl. The temperature of the mixture was maintained at $0 \pm 2^\circ\text{C}$.

Coupling:

The diazonium compound prepared as above was added slowly to the propyl hydroxylamine solution under constant mechanical stirring. The temperature of the reaction mixture was maintained at $0 + 02^\circ\text{C}$ throughout the reaction. By occasional addition of solid sodium acetate the pH of the reaction mixture was maintained between 5 to 6. The reaction mixture was further stirred for 15 minutes, after complete addition of diazonium compound. Sodium chloride was added in sufficient quantity to saturate the solution. Stirring was continued and after some time product comes out as orange coloured compound which was filtered under suction. The compound obtained is highly soluble in water so washing with water was avoided. Yield of the crude product was 6.1 gm. The crude compound was crystallised twice from water. The crystallised compound comes out as yellowish needle shaped crystals with melting point 298°C .

Elemental analysis:

	Molecular formula	$\text{C}_9\text{H}_{12}\text{N}_3\text{O}_4\text{SNa}$
	Theoretical value	Experimental value
%C	38.43	38.02
%H	4.30	4.23
%N	14.94	14.59
% Na	8.17	8.02

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