Short Communications

Synthesis of Propargyl Cellulose

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Propargyl cellulose has been synthesized by the allylation of cellulose followed by bromination and subsequent dehydrobromination with potassium tert-butoxide in dimethyl sulphoxide.

Unsaturated organic compounds having double or triple bonds are highly reactive and are useful as starting materials for synthesizing various derivatives. Introduction of unsaturation in the cellulose molecule has also received attention, because this can be used for imparting further chemical finishing treatments to cotton textile materials. This can be accomplished either by introducing a side chain containing unsaturation in place of one of the hydroxyl groups of the glucopyranose ring or by directly introducing a double or triple bond linkage on the glucopyranose ring itself.

Various cellulosic derivatives belonging to the above groups have been synthesized. However, cellulosic derivatives with triple bond between carbon atoms are difficult to prepare. Considerable work has been carried out in this field by Ragovin and his co-workers, who synthesized cellulose derivatives with a triple bond by different methods. Parker and Guthrie have described methods for preparing sodium propargyl sulphate and partially substituted propargyl ether of cellulose (in fibrous form) which involves treatment of the cellulose (cotton fabric) with sodium propargyl sulphate in sodium hydroxide.

Experimental Procedure

Preparation of allyl cellulose—Haller and Heckendorn method for preparing allyl cellulose, modified as per the suggestion of J.C. Arthur (Jr) (private communication), was used.

Cellulose (in the form of powder), after soaking in NaOH, was refluxed with allyl bromide. The degree of substitution of allylation was determined by estimating the bromine number.

Bromination of allyl cellulose—Allyl cellulose was treated with 0.4 N brominating solution in carbon tetrachloride for 48 hr at room temperature. The residual bromine was removed by washing the sample with alcohol.

Dehydrobromination of brominated allyl cellulose—Potassium tert-butoxide was synthesized in the laboratory by refluxing 20 g clean potassium metal in 500 ml freshly distilled tert-butanol under nitrogen pressure. When all the metal was dissolved, the excess of tert-butyl alcohol was removed by distillation until the crystals began to form. Finally, the white crystals of potassium butoxide were heated at 150°C at 0.1 mm pressure for 2 hr.

The dehydrobromination was carried out by treating the brominated allyl cellulose with potassium tert-butoxide in dimethyl sulphoxide for 48 hr at room temperature.

Estimation of triple bond—The presence of triple bond (C≡C) was inferred from infrared analysis by a weak absorption band around 2100 cm⁻¹. Another (C—H) absorption band was also observed around 3280 cm⁻¹ which is characteristic of hydrogen atom attached to C≡C.

Quantitative estimation of acetylenic hydrogen was carried out by the silver nitrate method. Silver nitrate (50 ml of 0.1 N) was taken in a 250 ml Erlenmeyer flask and 50 ml of 2% sodium acetate solution was added. The mixture was cooled in ice and 0.1 g sample of propargyl cellulose added. After shaking, the mixture was filtered and washed thoroughly with a minimum quantity of 0.01% sodium acetate solution. The filtrate was acidified with 5-6 ml conc. nitric acid and titrated against 0.1 N standard ammonium thiocyanate using ferric alum as indicator.
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References

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