

Synthesis of 1,3,5,5-Tetranitrohexahydropyrimidine with By-product of FOX-7 Prepared by 4,6-Dihydroxy-2-methylpyrimidine

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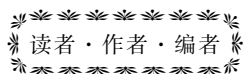
Abstract: By-product dinitromethane in the manufacturing process of 1,1-diamino-2,2-dinitroethylene (FOX-7) from 4,6-dihydroxy-2-methylpyrimidine (MPO) was recovered, the long needle dinitromethane potassium salt (KDNM) crystal with stable property was prepared via neutralization using aqueous potassium hydroxide solution, 1,3-dibutyl-5,5-dinitrohexahydropyrimidine was prepared via Mannich condensation reaction using KDNM, formaldehyde and *tert*-butylamine as raw materials, and 1,3,5,5-tetranitrohexahydropyrimidine (DNNC) was prepared via the nitrolysis of 1,3-dibutyl-5,5-dinitrohexahydropyrimidine with mixed acid system of concentrated sulfuric acid and concentrated nitric acid with total yield of 78.9% (Calculated by KDNM). The structure of DNNC and intermediate were characterized by ¹H NMR, IR, MS. The effect of pH value, solvents and temperature on Mannich condensation reaction, and the effect of selection of nitration system on the nitrating reaction were studied. The optimal conditions of Mannich condensation reaction were determined as: the molar ratio of KDNM, formaldehyde and *tert*-butylamine is 1.0 : 3.5 : 2.0, 10% aqueous methanol solution is used as solvent and hydrochloric acid is used to adjust pH value to 8 at room temperature; and then temperature rises up to 50 °C subsequently with reaction for 3 hours, and the yield reaches 85.3%. Using mixed acid composed of 20 mL 98% H₂SO₄ and 10 mL HNO₃ as nitrolysis system, the yield reaches 92.5%.

Key words: 1,1-diamino-2,2-dinitroethylene (FOX-7); dinitromethane potassium salt; 1,3,5,5-tetranitrohexahydropyrimidine (DNNC); Mannich reaction; nitrolysis reaction.

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