SYNTHESIS OF THE N-OXIDE OF 3-tert-BUTYL-6-METHYLURACIL

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Oxidation of 6-methyluracil by SeO₂ in boiling acetic acid leads to formation of the orotic aldehyde, which is not formed when other oxidizing systems are used (SeO₂ in water, conc. H_2SO_4 , dioxane, *tert*-butanol, CrO₃ in acetic acid and pyridine, H_2O_2 in alkaline medium and in the presence of iron salts) [1]. We have observed that upon oxidation of 3-*tert*-butyl-6-methyluracil by potassium permanganate in water, the methyl group in the 6 position is not oxidized, but rather the N-oxide of 3-*tert*-butyl-6-methyluracil is formed:



A solution of 15.8 g (0.1 moles) potassium permanganate in 300 ml water was added dropwise over the course of 30 min to a suspension of 18.2 g (0.1 moles) 3-*tert*-butyl-6-methyluracil in 100 ml water with stirring and cooling of the water. The stirring was continued for another 30 min, then the reaction mixture was filtered to remove the manganese oxide, acidified until the medium was neutral, evaporated under vacuum down to a volume of 50 ml. Then the shiny crystals formed were filtered off. Yield of the N-oxide of 3-*tert*-butyl-6-methyluracil, 8 g (40%). The product is stable during storage, and is easily soluble in aqueous bases and acids at pH 2, mp 193°C (from benzene). PMR spectrum (deuteroacetone): 9.84 (1H, s, OH); 6.01 (1H, s, H); 2.40 (3H, s, CH₃); 1.42 ppm (9H, s, C(CH₃)₃). In the mass spectrum of the oxide, among other peaks we see a low-intensity peak for the molecular ion with m/z 198 and an intense peak for the fragmentary ion with m/z 141 [M-t-Bu]⁺. Found, %: C 54.14; 54.24; H 7.08; 6.98; N 14.11; 14.18. C₉H₁₄N₂O₃. Calculated, %: C 54.30; H 7.08; N 14.14.

REFERENCES

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