Short Note

2-[(2-Hydroxy-naphthalen-1-ylmethylene)-amino]-5,6-dihydro-4H-cyclopenta[b] thiophene-3-carboxylic Acid Methyl Ester

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Abstract: Chitooligosaccharide with one 2,5-anhydro-D-mannofuranose unit at the reducing end (COSamf) was prepared by nitrous acid depolymerization of chitosan. The reducing-end functionalization of COSamf by reductive amination with octanoic hydrazide in the presence of NaBH₄CN was achieved in high yield. The chemical structure of the targeted octanoic hydrazide-linked COSamf was fully characterized by NMR spectroscopy and MALDI-TOF mass spectrometry. This synthesis opens the way to a new generation of COSamf derivatives with potential amphiphilic properties.

Keywords: 2-Aminothiophen, Anils, 2-hydroxy naphthaline, Schiff base

Schiff bases from 2-hydroxy-1-naphthaldehyde have often been used as chelating ligands in the field coordination chemistry [1]. The Schiff base compounds can be classified by their photochromic and thermochromic characteristics [2].

A solution of 2-Amino-5,6-dihydro-4H-cyclopenta[b] thiophene-3-carboxylic acid methyl ester 1 (5.0g, 0.025mol) and 2-hydroxynaphthaldehyde 2 (4.3g, 0.025 mol) in absolute ethanol (100 mL) was heated under reflux for 5 hrs. Cooling the mixture, filtering the precipitate and recrystallization from ethanol gave the Schiff base 3 as red crystals (6.4g, 72.88%).

Melting Point: 208-210ºC (EtOH).
IR (KBr; cm\(^{-1}\)): 1701.2 (C=O), 1560.6 (C=N), 1453.4 (C=C), 1308.4 (C-O) and 1046.2 (C-N).

\(^1\)H NMR (400 MHz; CDCl\(_3\)) \(\delta\) ppm: 14.93 (s, 1H, OH), 9.44 (s, 1H,=CH), 8.20- 7.27 (m, 6H, CH\(_{\text{aromatic}}\) 6H\(_{\text{aromatic}}\)), 3.99 (s, 3H,COOMe), 3.03-2.85 (t, 4H, 2 CH\(_2\)), 2.46-2.22 (m, 2H, CH\(_2\)).

\(^13\)C NMR (100 MHz; CDCl\(_3\)) \(\delta\) ppm: 27.50, 28.81, 29.78, 30.72, 49.69, 51.14, 54.05, 109.93, 118.92, 120.44, 123.17, 124.79, 127.40, 128.83, 130.32, 136.64, 146.45, 153.55, 158.86, 164.71.

References


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