

A FACILE SYNTHESIS AND CHARACTERIZATION OF LACTOSYLATED FORMAMIDINES NANOPARTICLES

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Abstract:

In view of application of Nanoparticles and desulfurized compounds of carbohydrates in industrial and medicinal research, we here by report the synthesized series of 1-Hepta -O-benzoyl - β -D-lactopyranosyl-3H/aryl formamidines nanoparticles and are characterized by IR, NMR and X-ray diffractions.

Index Terms: Dithiobiurets, Formamidines, Nanoparticles and characterization.

Introduction:

Desulfurization is the removal of sulfur or sulfur compounds (as from coal or flue gas), mostly from fuels. The most commonly required desulfurization process is natural gas, but it is also required for flue gas, coal and oil.

Sulfur in crude oil, natural gas, process gas and natural gas liquids (LNG) may take many forms, including hydrogen sulfide (H_2S), carbonyl sulfide (COS), sulfur oxide

(Sox) and the whole family of mercaptans. Raney nickel typically used in the reduction of compounds with multiple bonds, such as alkynes, alkenes, nitriles, dienes, aromatics and carbonyl containing compounds. Raney nickel is a spongy nickel, is a fine grained solid composed of mostly of nickel derived from a nickel-aluminium alloy. Several grades pyrophoric, most are used as air-stable slurries. Raney nickel is used as a reagent and as a catalyst in organic chemistry.

Similarly In view of this application¹ of lactosyl compounds and Nanoparticles in this we have synthesis to investigate the chemistry of this new compound with reference to their application.

Nanostructure materials are attracting a great deal of attention because of their potential for achieving specific processes and selectivity, especially in biological and pharmaceutical applications^{2,3}. Recent studies have demonstrated that especially formulated nanoparticles have good antibacterial activity^{4,5}.

Experimental:

UV-visible Spectra is measured using UV Spectrophotometer by using model Single Beam UV-Visible Spectrophotometer with software(BI/CI/SP/SB-S-03)of Bio Era make.. IR spectra were recorded on Perkin-Elmer spectrum RXI FTIR spectrophotometer ($4000-450\text{ cm}^{-1}$). 1H NMR was recorded in $CDCl_3$ on Bruker DRX-300 spectrometer operating at 300 MHz.

a) Synthesis of hepta-O-benzoyl- α -D-lactosyl bromide:

The finally powdered lactose octabenzoate(0.03M, 21.0g) was added gradually to the brominating agent. After the addition the flask was kept for 2hr at room temperature. Then the reaction mixture with chloroform (130ml) then the mixture was shaken vigorously for about 15 min. The resultant mixture was poured into ice cold water. The chloroform layer was then separated. It was washed several with aqueous sodium bicarbonate to remove excess of acetic acid followed by aqueous sodium metabisulphite to remove excess of bromine and finally 2-3 times with water. To the chloroform addition of petroleum ether afforded a solid (16.5 gm). This solid was expected hepta-O-benzoyl- α -D-lactosyl bromide (yield 77%). It was purified by dissolving it in minimum quantity of chloroform and reprecipitating it with petroleum ether, m.p. 168⁰C.

b) Preparation of lead thiocyanate :

Lead thiocyanate was prepared by mixing aqueous solution of lead nitrate and ammonium thiocyanate. The white granular lead thiocyanate was filtered washed with distilled water and dried at 50⁰ C.

c) Preparation of hepta-O-benzoyl- β -D-lactosyl isothiocyanate⁶ :

To a suspension of hepta-O-benzoyl- α -D-lactosyl bromide (21 gm, 0.03M) in sodium dried xylene (80ml) was added lead thiocyanate (6gm, 0.03M). The reaction mixture was then treated for microwave synthesis for about 3 min. This solution was then cooled and liberated lead bromide was removed by filtration. The xylene filtrate was then treated with petroleum ether (60-80⁰C) with stirring, a white solid mass obtained (13gm). This solid was expected hepta-O-benzoyl- β -D-lactosyl isothiocyanate.

It was purified by dissolving it in minimum quantity of chloroform and reprecipitating it with petroleum ether, m.p. 118-120⁰C. [Found; C; 67.07, H; 4.46, N; 1.22, S; 2.9; C₆₂H₄₉O₁₇NS requires; C; 66.96, H; 4.41, N; 1.26, S; 2.88%].

Preparation of 1-hepta O-benzyl - β -D -lactosyl 5 phenyl 2,4,Dithiobiurets:

A suspension of 4 gm of Hepta O-benzyl- β -D lactosyl isothiocyanate with 20 ml of benzene and 1 gm of aniline thiourea was treated for microwave synthesis for about 3 min. This solution was then cooled and the benzene filtrate was then treated with petroleum ether (60-80⁰C) with stirring, a white solid mass obtained (13gm). This solid was expected 1 -hepta-O- β -D lactosyl 5-phenyl 2, 4 dithiobiurets.

It was purified by dissolving it in minimum quantity of chloroform and reprecipitating it with petroleum ether, m.p. 145-146⁰C.

Desulphurization of Hepta-O-benzoyl- β -D-lactosyl-5-aryl-2,4dithiobiuret

1. Preparation o Raney Nickel:

The required Raney nickel was prepared by earlier method ²⁹ by action of sodium hydroxide solution on powdered NI-Al alloy.

Preparation o 1-Hepta-O-Benzoyl –D-lactopyranosyl-3-H/aryl formaides:

Preparation of Nanoparticles 1-Hepta- O-benzyl – β -D-lactosyl- 5-phenyl 2,4 Dithiobiurets

Take about 1 gm of 1-Hepta-O-benzyl – β -D-lactosyl -5-phenyl 2, 4 Dithiobiurets and dissolve complete 1 Hepta O-benzyl – β -D –lactosyl- 5 –phenyl 2,4 Dithiobiurets in the 50ml of solvent in 250 ml beaker. Now put this beaker in sonicator. The highly penetrating acoustic waves are passed through mixture, which create high pressure bubbles in **the beaker due to which breakdown of the bulk material is takes place and desired sized** nanoparticles are formed. The size determination of nanoparticles is done by the X-ray diffraction studies.

IR spectrum of 1-Hepta- O-benzyl – β -D-lactosyl- 5-phenyl 2,4 Dithiobiurets⁷

Absorption Observed (Cm ⁻¹)	Assignment	Absorption Expected (Cm ⁻¹)
3068	C-H Ar-stretching	3040-3010
1728	C=O stretching	1750-1735
1176	C-O stretching	1210-1153
1026, 909	Characteristic of lactose	1100-1000 and 910-900
710	Monosubstituted benzene	770-680

NMR SPECTRAL STUDIES^{8,9}:

The NMR Spectrum of compound distinctly displayed signals due to N-H Proton at δ 9.05 and d 6.57 ppm, Aromatic Protons at δ 7.47-7.15 ppm, lactosyl protons at d 5.77-3.76 ppm.

Characterization of Nanoparticles:

- Charterisation using UV – Visible Spectrophotometer:** Characterization of nanoparticles was done using visible Spectrophotometer by using model Single Beam UV-Visible Spectrophotometer with software (BI/CI/SP/SB-S-03)of Bio Era make. The UV-Visible Spectroscopy reveals the formation of nanoparticles by showing different absorption those from bulk material.
- Size determination of Lactose Octabenzoate Nanoparticles by X-Ray Diffraction Studies:** From the X-Ray diffraction it comes to know that size of nano octabenzoate is 38nm.

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