Synthesis 3-(2-propyne-OXY)-1,2: 6,6-di-O-isopropylidene-α-D-gluco ranse and react with secondary amine ((Ma nich reaction))

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M.N .Mohamad College of Agriculture . Dep.of solid
Al-Qadisiah Uni
E.mail : moyyad2001 @yahoo.com

Abstract

The aim of the present work is the synthesis of acetylen sugar, having acetyten group at position (3) the acelyenlce either derirative was left to react with secondary amine in the presence para formaldehyde (mannich - reaction)

Chemical Classification QD 241-441
Key words : acetylene sugar , proargyl bromide , secondary amine .

Introduction:

The diacetone glucose (1) was the starting meterial for this synthesis which was then treated with propargyl bromide in the presence tetra butyl amonium bromide to yeild successfully 3-(2-propynyl - oxy) -1,2 5, 6 – di- O – isopropylidene –a-D- glugofurance (2) , was then left to react with secondary amines in the presence of para formaldehyde (mannich –reaction) , the position (3) have been found to display a wide ranye of biological activates .

Experimental:

Unless otherwise stated , the following generalizations apply IR. spectra were measured in pye unica 9712 spectro photometer in nujol . H n.m.r spectra were measured with Hitaha R-24 B at 90MHz0 . in CDCI3 with TMS as internal standard . Thin layer chromato graphy were measured with whatman silica gel F 254 .

Experiment (1) :

Preparation of 1,2: 5, 6 - di - 0 -- isopropylidene – α-D-lucofuranse Glucos anhydride (50m mole) in crystallization by chloroform and hexane (2:1) m.p (107-109C) .
T.L.C. (benzene - methanol 8: 2)

acetone analar (500ml) and shaking the mixture (10 minutes) ,added ferric' chloride anhydrous (18 m mole) , The reaction mixture was refluxed on a water bath (10 hr) . then added potassium carbonate anhydrous (10%) neutralized and acid reaction-extraction of diacetone glucose by chloroform and crystallization by chloroform and hexane (2:1) m.p (107-109C) .
T.L.C. (benzene - methanol 8: 2)

experiment (2) :

Preparation 3-(2-propynyl - oxy) = 1,2: 5, 6-di-0–isoproylidene- α , D- gluco furanose (2) .

Diacetone glucose (1) (10 mmole] in benzene 50 ml added sodium hydroxide (20% gm) , drops of proparyl bromide (10mmole) and tetra tutyl bromide. The reaction mixture was refluxed on a water bath until the evolution-of carbon dioxde-(12 hr) .

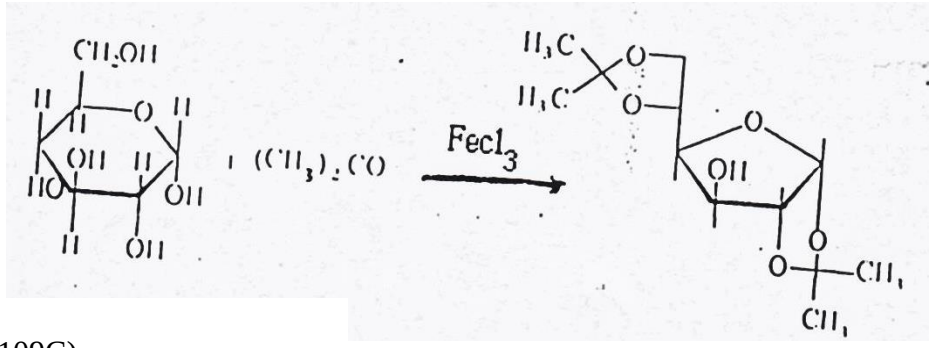
The product were purifiet from benzene precipitate (viscous
liquid TLC (benzene - methanol 9=1) .

Experiment (3) :

3 -(4 - (N, N - diethyl amins) - but -2-ynyloxy) -1,2 = 5,6 -di - O – isopropylidene-α-D- gluco furanose . (3)

Mannich Reaction (4,5) :

Derivative (2) (10 mmole in 50 ml dioxane added para formaldehde (10 mmole) shaking the mixture (5 minutes) - was then left to react all the analysis carried out fit the product obtained.



m.p = (107-109C)

Aual –calc for C12 H2O O6 = C, 55. 38, H , 7.69

Found = C , 55 , H. 7 .69

1R	group	max cm-1
	OH	3400

n.mr

DMSO-D6) H-1 δ =5.35 , d)

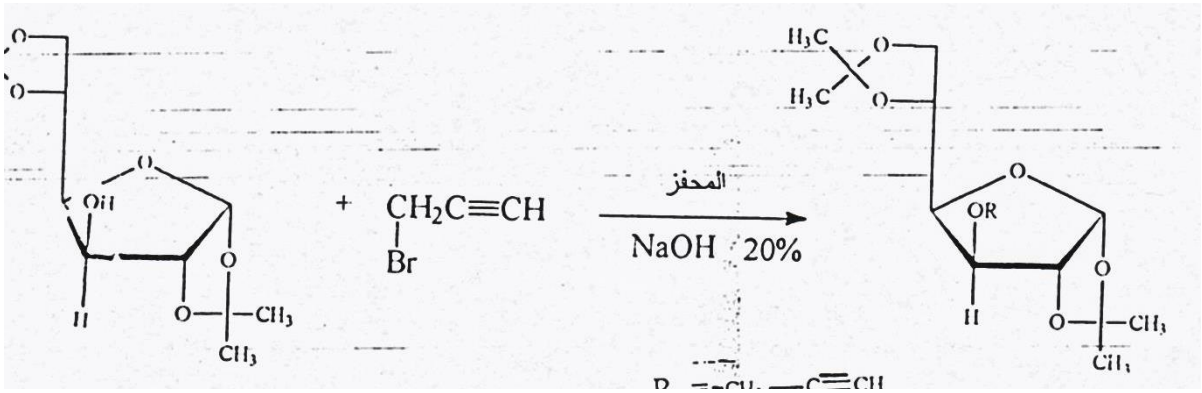
H-2 (δ= 4-40d)

OH , H-3 , H-4 , H-5 (δ =3.60-4.2m)

(2 (H-6) (δ -0.9 – 1.3)

(4 × CH3) (δ =(0.8-1) 124 Triplet

The product (2) obtained from :



Anal cale for C15 H22 O6 = C , 60 -40 , H0 7.60

Found = C , 59 – 30 , H , 7-57 .

1 R group CH≡C-1200 cm-1

n.m.r (DMSO- D6)

(H-1) (δ = 5.50 , d)

(H-2) (δ = 4.30 , d)

(H-3 , H-4 , H-5) (δ =3.70-400M)

n.m.r (DMSO- D6)

(H-1) (δ = 5.50 , d)

(H-2) (δ = 4.30 , d)

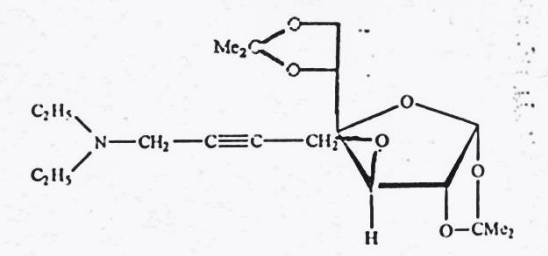
(H-3 , H-4 , H-5) (δ =3.70-400M)

(0-CH2-C ≡ C) (δ= 2-15 ,d)

(≡ C-H) (δ = 2.85T)

(4 × CH3) (δ= 0.9-1.1) 12HT)

The product (3) obtainal from



References:

1. M.N. Mohamad, 2004,J.Al Marj. Libya for pure science,Vol,5,No 3.
2. M.N.Mohammad,2005, J-Al-Qadisiah for pure science, Vol 10, No.1
3. M.H.Klemens,s.p.chon and R. H. weld 1996,J.Med. Chem. 35. 1212.
4. D,J. sheehan, c.d., vol.66 1993, clin. infect Dis., 17, 494.
5. K.Krolova, 1992, J-chem. Pap 25, 266.
6. K.Krolova, 1992, j-chem pap, 52, 52-
7. D.E.Snider, 1998, New, Engl. J.Med., 338, 1689.
8. D.A.Mitchison, 1996, Natur. Med.,2,6
9. MODolezal, M.Miletin. J .Kunes and K.Krolava 2002, molecules , 7 , 363-373.
10. R.Grigg, 1987,.J.Chem.soc. che,commun.49.

تحضير 3 (2-بروباين- اوكسي) -1 ، 2 :5 ، 6-ثنائي-O – ايزوبروبلين –الفا- Dكلوكوفيور انوز وتفاعله مع امين ثانوي (تفاعل مانخ)

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د.مؤيد نعمة محمد– جامعة القادسية / كلية الزراعة / قسم علوم التربة

الخلاصة :

ان الهدف من البحث تحضير سكر استيلين فتكون مجموعة الاستيلين في الموقع 3 ثم مفاعلة هذا السكر مع الامين الثانوي بوجود بارافورماليدهايد ويسمى هذا التفاعل (تفاعل مانخ) .

كلمات مفتاحية : سكر استيليني ، بروباين بروميد ، امين ثانوي