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Some Transformative Reactions of Odollactone

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Abstract Oxidative rearrangement of odollactone has been carried out with hydrogen peroxide containing *p*-toluene sulphonic acid in dichloromethane. The products obtained were characterised by chemical transformation using selenium dioxide in acetic acid, sodium dichromate in acetic acid and Li in ethylenediamine followed by spectral analysis. The introduction of olefinic double bond at AB-ring juncture influences the cleavage of γ-lactone ring to the diol in contrast to the parent odollactone that gives the carboxylic acid.

Keywords Wagner Rearrangement, Friedelane Skeleton, Thermodynamically Stable, Heteroannular System, Li In Ethylenediamine

1. Introduction

Although structure (**1**) for odollactone[1,2] has been characterized[3-6] from our laboratory from the plant *Gynocardia Odorata* available in the hill area of Darjeeling, detailed investigation in respect of its chemical activity has not yet been attempted so far. Keeping this view in mind and to explore the chemical activity of this naturally occurring 3β-hydroxy lactone of friedelane skeletone, we have studied some oxidative and reductive transformations on it. During the investigation some interesting observations were noted which comprise the subject matter of this report.

2. Result and Discussion

It has been reported[7] that friedelanol when heated with *p*-toluene sulfonic acid, undergoes rearrangement of the friedelane skeleton to oleanane skeleton via Wagner rearrangement[8]. Odollactone has a γ-lactone function attached to the C-13 and C-15 atoms of friedelane skeleton, thus it is anticipated that movement of the carbocation is rather impossible beyond C-14 position as the migration of C-27 carbonyl carbon from C-13 to C-14 position will give rise to the formation of highly strained β-lactone. Therefore, it was assumed that the carbocation generated at C-3 will have a restricted migration up to C-5 position giving rise to olefinic double bonds at C-5(6)/5(10)/1(10) of glutene[9] derivative (**2**) or it may further migrate up to C-9 forming double bond at C-8(9)/C-9(11)/ or C-7/(8) of multiflurene/baurene[10] derivative (**3**). Under the strongly acidic condition the formation of thermodynamically more stable tetrasubstituted olefins at C-5(10) or C-8(9) position is more likely (isomer **2**or **3**).

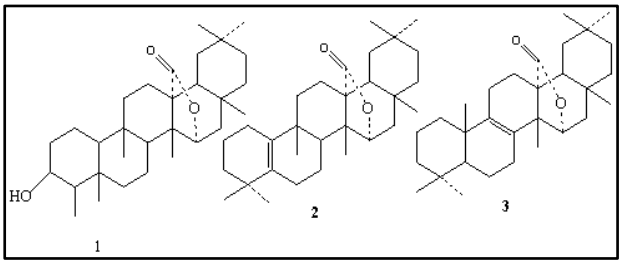

Figure 1. Structure of odollactone (**1**), glutene derivative (**2**), multiflurene/baurene derivative (**3**)

In order to examine the above possibilities, (**1**) was subjected to the treatment with *p*-toluene sulfonic acid and H_20_2 in ^{t}BuOH (vide infra)[11-13]. The compound obtained after usual workup and crystallization from chloroform-methanol furnished a crystalline solid (**A**), m.p. 285 °C. It was analysed for $C_{30}H_{46}O_2$. It responded to positive TNM test indicating the presence of olefinic double bond. IR absorption at 1775 cm^{-1} showed the existence of γ-lactone in (**A**). This observation shows that the lactone ring is stable enough to undergo hydrolysis under the acidic condition; it also reveals that the lactone ring remains intact in its position. Mass spectrum showed the molecular ion at m/z 438 [M^+], the fragments at m/z 423 was the base peak formed due to the loss of a methyl group from the molecular ion. The other notable peaks appeared at m/z 377, 353, 341, 257, 255, 239, 201, 189, 175, and 123.

PMR spectrum of the compound (**A**) showed the presence of seven tertiary methyls at δ 0.92 (2 x Me), 0.94, 0.95, 0.97, 1.01 and 1.15 ppm, the lactyl proton appeared at its usual position at δ 4.32 ppm as a triplet with a small J value of 3 Hz. A comparison of the position of methyl groups in the deoxylactone[14,15] (**2**) indicated that the methyls at δ 1.15, 0.95 and 0.97 are not shifted and hence indicated that the gem dimethyl group at C-20 carbon and the angular methyl at C-17 position remains intact in forming compound (**A**). The absence of olefinic proton indicated that the newly

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formed double bond is void of vinyl proton and must be tetra substituted one.

The ^{13}C NMR spectrum of (**A**) indicated the presence of seven tertiary methyls at δ 13.7, 19.4, 27.9, 28.7, 29.7, 31.3 and 34.3 ppm as quartets; eleven methylene carbons at δ 19.8, 20.5, 22.2, 24.6, 26.4, 31.7, 34.1, 34.2, 35.9, 39.5, 40.5 ppm as triplet; three doublets at δ 43.7, 44.0 and 80.8 ppm. The last one (80.8 ppm) being due to the carbon bearing the lactyl oxygen at C-15 position and nine singlets of which six peaks at δ 28.1, 30.4, 33.8, 36.8, 47.8 and 50.9 ppm were due to sp^3 carbons, the lactone carbonyl carbon appeared at δ 180.3 ppm and the carbon bearing the double bonds resonate at δ 133.3 and 136.1 ppm.

From the above data it is obvious that compound (**A**) has acquired a tetra substituted double bond which must be either at 5(10) or 8(9) position giving rise to the structure of (**A**) either as (**2**) or (**3**) respectively.

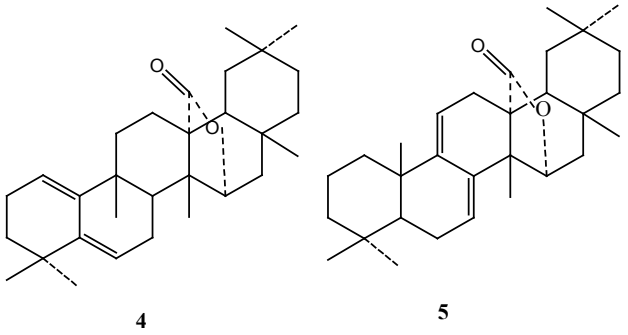
In order to establish the structure of compound (**A**) as either (**2**) or (**3**) the following chemical reactions were carried out:

(a) Oxidation with Selenium dioxide in acetic acid Compound **A** was subjected to the reaction with selenium dioxide in acetic acid under reflux condition. After usual work up and purification by chromatography and repeated crystallizations from chloroform-methanol a crystalline solid (A-1) of m.p. 295-96 ℃, $[\alpha]_D$ +75° was obtained. UV spectrum of (**A-1)** showed two absorption peaks at λ_{max} 233 and 237 nm suggesting that the diene is in a heteroannular system. Its IR spectrum showed absorption peaks at 1760 cm^{-1} and 820 cm^{-1} indicating the presence of carbonyl group of γ-lactone ring and trisubstituted double bond respectively. Mass spectrum of (**A-1)** showed molecular ion at m/z 436 [M^+], with other fragments at m/z 421, 375, 287, 239, 213, 202, 173, 145 and 134 (base). The PMR of this compound showed singlets for seven tertiary methyls at δ 0.91 (2 x CH_3), 0.96, 0.97, 1.04, 1.08, 1.18 ppm indicating that C-4 contains two geminal methyl groups; the C-15 lactyl proton appears at its usual position at δ 4.26 ppm with small J value of 3 Hz and two olefinic protons at δ 5.4 and 5.51 ppm as triplets with J = 3 Hz that coupled with protons centred at δ 2.1 ppm (appeared as multiplets).

^{13}C NMR spectrum of (**A-1)** showed the presence of 30 carbons, seven tertiary methyls appeared as quartets at δ 14.1, 21.1, 26.5, 28.6, 30.2, 32.4, 33.7; eight methylene carbons as triplets at δ 22.6, 23.1, 24.3, 32.6, 33.9, 34.6, 36.3, 40.5; five -CH carbons, two as doublets at δ 41.0, 43.7 of saturated carbons; one containing the lactyl oxygen appeared at δ 80.8 ppm and two olefinic carbons at δ 116.4 and 117.9 ppm; eight singlets, six of which are due to saturated sp^3 carbons appearing at δ 28.1, 30.1, 33.2, 35.3, 47.2, 51.4 and two as olefinic carbons at δ 141.1 and 144.3 and a single carbonyl carbon at δ 179.5 ppm.

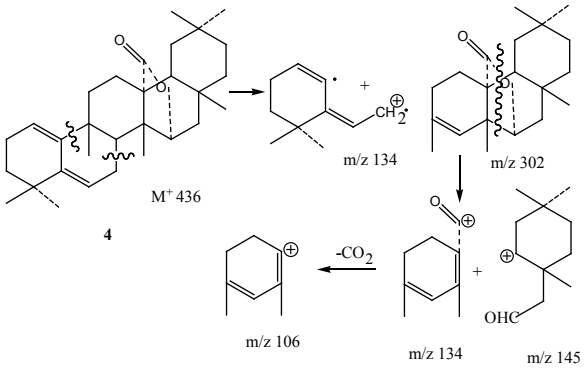
Thus from the above spectral data, the structure of the compound (**A-1**) is likely to be either glut-1(10),5(6)-diene (**4**) or multifluor-7(8),9(11)-diene derivative (**5**).

However, the UV spectrum of (**A-1**) is found different from that of compound having conjugated diene with double bonds at C-7, C-9(11) when compared with compounds reported earlier[10]. Thus it is presumed that compound (**A-1**) has its double bonds at 1(10), 5(6) position resulting in the formation of diene (**4**) rather than (**5**). This is further supported by the mass fragmentation pattern of (**A-1**), which showed base peak at m/z 134 by the rupture of ring B as shown in the Scheme 1. This can easily be explained by considering the double bonds at 1(10) and 5(6) position.



4 **5**

Figure 2. Structure of glut-1(10),5(6)-dien-27→15α olide (**4**) and multifluor-7(8),9(11)-diene-27→15α olide (**5**)



Scheme 1. Mass fragmentation of compound (**4**)

(b) **Sodium dichromate oxidation** Compound (**A**) dissolved in acetic acid was refluxed with sodium dichromate. The product obtained after usual work up and purification on chromatography and crystallization furnished crystalline solid (**A-2**) of m.p. 316℃, $[\alpha]_D$ +16°. Elemental analysis of the compound (**A-2**) indicated its molecular formula as $C_{30}H_{44}O_4$. IR spectrum of the compound showed absorption peaks at 1750 and 1720 cm^{-1} showing the existence of γ-lactone ring and a six membered ketone functions. (**A-2**) showed negative TNM test for olefinic double bond and its UV spectrum showed humps at λ_{max} 250 and 290 nm for the carbonyl functions. Mass spectrum of (**A-2**) showed the molecular ion at m/z 468 [M^+], with base peak at m/z 123 including the other fragments appeared at m/z 453,440, 425, 407, 371, 315, 255, 233, 217, 206 and 153.

The PMR spectrum of compound (**A-2**) showed presence of seven tertiary methyls at δ 0.99, 1.0 (2 x -CH_3), 1.06, 1.19, 1.20, 1.27; a triplet at δ 4.17 (J = 3Hz) was due to the C-15 lactyl proton; a doublet of a doublet at δ 2.39 with coupling

constant of 7 Hz and 3 Hz and another at δ 2.16 with J = 7 Hz and 15 Hz which is due to CH_2 protons adjacent to the carbonyl group, these two protons couple with a lonely proton at δ 1.89 as a triplet with J =7 Hz. These observations assigned the position of the keto group at C-6.

^{13}C NMR of (**A-2**) exhibited resonating peaks for all the thirty carbons, seven of them are quartets and appeared at δ 13.3, 16.7, 23.5, 25.8, 30.0, 31.9, 33.3; nine triplets at δ 17.1, 32.2, 29.3, 33.9, 34.5, 35.9, 36.5, 37.2, 40.2; three doublet at δ 34.9, 43.9 and 80.5 and ten singlets, six of them being due to sp^3 tertiary carbons at δ 28.1, 30.2, 31.5, 36.9, 47.2, 51.0 and two at δ 69.6 and 73.1 are due to carbons attached to the oxygen atom and the two downfield carbons at δ 178.8 and 206.1 ppm are due to the carbons of lactone carbonyl and the C-6 ketone functions respectively. From the foregoing spectral data the structure of the compound (**A-2**) has been assigned as (**6**).

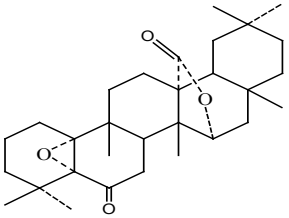


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Figure 3. Structure of glut-5α,10α-epoxy-27→15α-olide (**6**)

(c) **Reduction with lithium in ethylenediamine** The lactone (**2**) on reduction with lithium in ethylenediamine in the usual process[16-18] furnished a crystalline solid (**A-3**) of m.p. 265-66 °C. Elemental analysis indicated its molecular formula as $C_{30}H_{50}O_2$. It showed the presence of hydroxyl groups in its IR spectrum with a broad band ranging between 3200–3400 cm^{-1} indicating that the lactone ring of (**2**) has been cleaved to the hydroxyl groups. Mass spectrum of the compound showed the molecular ion peak at m/z 442 [M^+] with other fragments at m/z 424 [M^+ - H_2O], 409 [424 – CH_3], 393 [424–CH_2OH] (base peak), 379, 255, 231, 189, 175, 149, 123, 107, 95, 81, 69. PMR spectrum of the compound showed a broad band at δ 4.08 ppm for the C-15 proton. A double doublet appeared at δ 3.41 ppm with J value of 12 Hz each for the geminal protons of the C-27 carbon having the group CH_2OH; it also showed the presence of seven tertiary methyls between δ 0.97 to 1.25 ppm. ^{13}C NMR showed the presence of all the thirty carbons; seven as quartets, twelve as triplets and the triplet at δ 64.6 was due to the carbon attached to the hydroxyl group, three doublets at δ 41.3, 43.1 and 72.2, the last one being that -CH carbon containing the hydroxyl group and may be assigned to the C-15 carbon (CHOH); eight singlets at δ 28.3, 29.1, 33.8, 38.0, 42.2, 43.3, 132.6 and 137.9 (the last two signals being due to olefinic carbons) ppm. Thus ^{13}C NMR signals confirmed that Li in ethylenediamine converts the lactone (**2**) to a diol (**7**) with complete rupture of the lactone ring without forming the other possible reduction products *viz* the carboxyl group. This reduction with Li in ethylenediamine that results in the rupture of lactone ring with the formation of diol is in contrary to the usual product of cleavage at the lactyl O-C bond forming the carboxylic group[7].

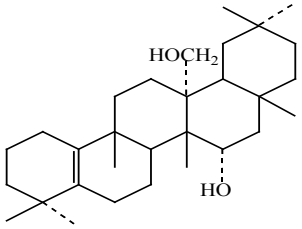

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Figure 4. Li/EDA converts lactone (**2**) to a diol (**7**)

3. Experimental

Materials and Method

Melting points are uncorrected. Petroleum had b.p. 60-80°C. PMR spectra (chemical shifts in δ, ppm; TMS as internal standard) and ^{13}C NMR spectra were recorded in $CDCl_3$ Bruker avance 300 MHz FT NMR spectrometer, Brucker WH-400 and Brucker WH-270 with DEPT programme respectively; IR spectra in nujol on Beckman IR-20 and Perkin-Elmer FTIR spectrophotometer using KBr discs; UV spectra in methanol on Shimadzu UV-240 spectrometer; mass spectra in Varian MAT 711 instrument at 70 eV. All the chemicals were reagent grade (from Merck, Fluka, SRL) and were purified prior to their use following the standard protocols. Column chromatography was performed over silica gel. TLC was performed over silica gel G (BDH 60-120 mesh).

3.1. Treatment of Compound (1) with H_2O_2-*p*TsOH

To a solution of (**1**) (2.0 g) in CH_2Cl_2 (100 mL) was added to a solution 80 mL prepared by mixing *p*-TsOH (3 g) and 30% H_2O_2 (5 mL) in ^{t}BuOH (80 mL). The mixture was stirred slowly for 24 hrs and poured into water. It was then extracted with CH_2Cl_2, washed with water, dried by Na_2SO_4 and the solvent removed under reduced pressure. The residue (1.5 g) was purified by column chromatography over neutral silica gel. Crystallization from $CHCl_3$-MeOH furnished a crystalline solid (**A**), m.p. 285 °C. The compound (**A**) was analyzed for $C_{30}H_{46}O_2$. It responded positive to TNM test indicating the presence of olefinic double bond. IR: 1775 cm^{-1}. MS: m/z at 438 [M^+], 423 (base peak) [M^+-CH_3] and other peaks are m/z 377, 353, 341, 257, 255, 239, 201, 189, 175 and 123. PMR: δ 0.92-1.15 ppm (7 x t-Me protons), 4.32 ppm lactyl proton of C-15 (t, 1H, J=3Hz). ^{13}C NMR: seven t-Me groups at δ 13.7, 19.4, 27.9, 28.7, 29.7, 31.3 and 34.3 ppm as quartets; eleven methylene carbons at δ 19.8, 20.5, 22.2, 24.6, 26.4, 31.7, 34.1, 34.2, 35.9, 39.5, 40.5 ppm as triplet, three doublets at δ 43.7, 44.0 and 80.8 (C-15 lactyl oxygen) and nine singlets of which six peaks at δ 28.1, 30.4, 33.9, 36.8, 47.8 and 50.9 ppm were due sp^3 carbon, at δ 180.3 ppm (lactone carbonyl carbon), at δ 133.3 and 136.1 ppm for double bond.

3.2. SeO_2 Oxidation of Compound (2)

Compound (**2**) (200 mg) was refluxed in glacial acetic acid (30 mL) with SeO_2 (150 mg) for 3 hrs. After usual work up and purification by chromatography and repeated crystallization from $CHCl_3$-MeOH a crystalline solid (**A-1**) was obtained. The molecular formula of the compound is $C_{30}H_{44}O_2$. m.p: 295-96 ℃, $[\alpha]_D$ +75°. UV: λ_{max} at 233 nm and 237 nm; IR: 1760 cm^{-1}, 820 cm^{-1}. MS: m/z 436 [M^+], other fragments at m/z 421, 375, 287, 239, 213, 202, 173, 145, 134 (base). PMR: six singlet for seven tertiary methyl at δ 0.9 (2-CH_3), 0.96, 0.97, 1.04, 1.08, 1.18 ppm; δ 4.26 ppm (t, 1H, J=3 Hz) and δ 5.4 and 5.51 ppm as triplet with J = 3 Hz for two olefinic protons. ^{13}C NMR: Seven tertiary methyls appeared as quartet at δ 14.1, 21.1, 26.5, 28.6, 30.2, 32.4, 33.7; eight methylene carbons as triplets at δ 22.6, 23.1, 24.3, 32.6, 33.9, 34.6, 36.3, 40.5; five CH carbons as doublets at δ 41.0, 43.7, 80.9, 116.4 and 117. 9 and eight singlets six of which saturated sp^3 carbons appearing at δ 28.1, 30.1, 33.2, 35.3, 47.2, 51.3 and two as olefin carbon at δ 141.1 and 144.3 ppm and a single carbonyl carbon at δ 179.5 ppm.

3.3. Sodium Dichromate Oxidation of Compound (2)

A solution of $Na_2Cr_2O_7$ (5 g) in glacial acetic acid (150 mL) was added slowly during a period of 1 hr to a vigorously stirred solution of product (**2**) in refluxing CH_2Cl_2 (50 mL). The mixture was refluxed for 24 hrs and then cooled. Excess dichromate was decomposed with EtOH (50 mL). The solution was concentrated to 1/3 rd volume and the content poured into ice cold water, extracted with ether, washed with water and dried (Na_2SO_4). Removal of solvent and chromatography of the residue furnished a solid which on crystallization gave the compound (**A-2**). m.p: 316 ℃, $[\alpha]_D$ +16°. The molecular formula of the compound is $C_{30}H_{44}O_4$. IR: 1750 & 1720 cm^{-1} (γ-lactone ring & a six membered ketone). TNM test is negative. UV: λ_{max} at 250 nm and 290 nm. MS: m/z 468 [M^+], the other fragments appeared at m/z 453, 440, 425, 407, 371, 315, 255, 233, 217, 206, 153 and 123 (base peak). PMR: δ 0.99, 1.0 (2 x-CH_3), 1.06, 1.19, 1.20, 1.27 (s, 7xt-Me), δ 4.17 (t,1H, J = 3 Hz) due to C-15 lactyl proton, δ 2.39 (dd,J = 7 Hz and 15 Hz) and δ 2.16 (dd, J =7 Hz and 15Hz) for CH_2 proton adjacent to the carbonyl group, these two protons couple with a lonely proton at δ 1.89 (t, 1H, J = 7 Hz). ^{13}C NMR: Seven tertiary groups as a quartet at δ 13.3, 16.7, 23.5, 25.8, 30.0, 31.9, 33.3; nine triplets at δ 17.1, 32.2, 29.3, 33.9, 34.5, 35.9, 36.3, 37.2, 40.1; three doublet at δ 34.9, 43.9 and 80.5 and ten singlets six of them being due to sp^3 tertiary carbons at δ 28.1, 30.2, 31.5, 36.9, 47.2 and 51.1, two at δ 69.6 and 73.1 are due to carbons attached to the oxygen atom and the two resonating down field carbons at δ 178.8 and 206.1 ppm are due to the carbons of lactone carbonyl and the (C-6) ketone functions.

3.4. Reduction the Compound (2) with Lithium in Ethylenediamine

Lactone (**2**) (200 mg) was refluxed in ethylene diamine (150 mL) with lithium metal (200 mg) in presence of nitrogen atmosphere for 3 hrs. After usual work-up and purification by chromatography and repeated crystallization from $CHCl_3$-MeOH, a solid (**A-3**) was obtained. The molecular formula was analysed $C_{30}H_{50}O_2$, m.p: 265-266 ℃, IR: broad band ranging between 3200-3400 cm^{-1} for hydroxyl group. MS: m/z 442 [M^+], 424 [M^+-H_2O], 409 [424-CH_3], 393 [424- CH_2OH] base peak, other fragments appeared at m/z 379, 255, 231, 189, 175, 149, 123, 107, 95, 81, 69. PMR: δ 4.08 (for C-15 proton), δ 3.41 (dd, J = 12 Hz for gem-protons of C-27). Seven tertiary methyl groups appeared at δ 0.97 (2xCH_3), 1.0 (3xCH_3), 1.02 (CH_3), 1.25 (CH_3). ^{13}C NMR: Seven quartets for seven CH_3 group at 20.0, 21.2, 28.0, 29.0, 30.3, 33.5, and 35.7; twelve triplets at δ 19.9, 20.1, 24.5, 26.3, 28.7, 31.9, 32.5, 35.9, 39.1, 39.7, 48.1 and 64.5 (CH_2OH). Three doublets at δ 41.3 43.1, 72.2 (at C-15 as CHOH); eight singlet's at δ 28.3, 29.1, 33.8, 38.0, 42.2, 43.3, 132.6 and 138.0 ppm (last two signals being due to olefinic carbons).

4. Conclusions

From the above chemical reactions involving oxidative and reductive transformations of the rearranged product (**2**), it is evident that usual migration of carbocation to give the most stable olefinic products like β-amyrene[7] or δ-amyrene is restricted to the C-5(10) position due to the presence of lactone ring at C-27(15α) position. It is further concluded that the presence of olefinic double bond at AB- ring juncture (5/10) position facilitates the formation of diol rather than the carboxylic group as observed in the case of parent compound odollactone[7].

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