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SYNTHESIS OF FUNCTIONALISED BICYCLIC α-METHYLENE-γ-BUTYROLACTONES FROM 2,3-CYCLOHEXENE ACETAL VIA RADICAL CYCLISATION APPROACH[1]

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Abstract: *2,3-Cyclohexene acetal, wherein the acetal functionality is derived from 2R,3R(+)-tartaric acid, has been transformed regioselectively into functionalised bicyclic α-methylene-γ-butyrolactones via radical cyclisations. The radicals were derived from n-Bu$_3$SnH reactions with secondary bromides and a tertiary nitro compound.*

We have recently reported on the conversion of 2,3-olefinic acetals **1** into 3-nitro-2,3-olefinic ketones[2] **3** and 1,2- and 1,3-diones[3] **5** and **7** respectively. Presence of the acetal moiety was found to exhibit marked regioselectivity on these reactions. The intermediates **2** and **6** (scheme 1) have been transformed into useful intermediates by us[4]. α-Methylene-γ-butyrolactones are found in a number of biologically important natural products and excellnt reviews of their synthesis have recently appeared[5].

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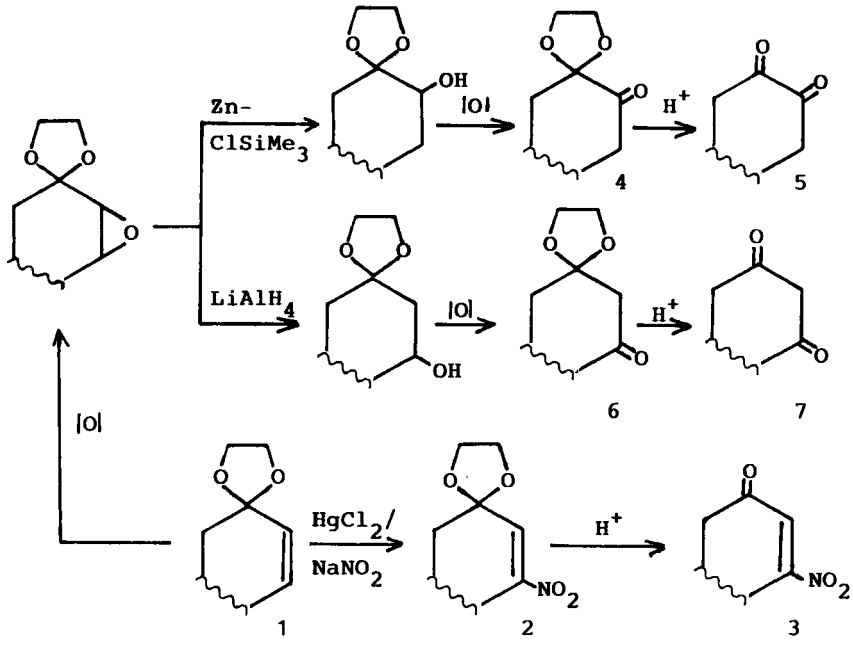


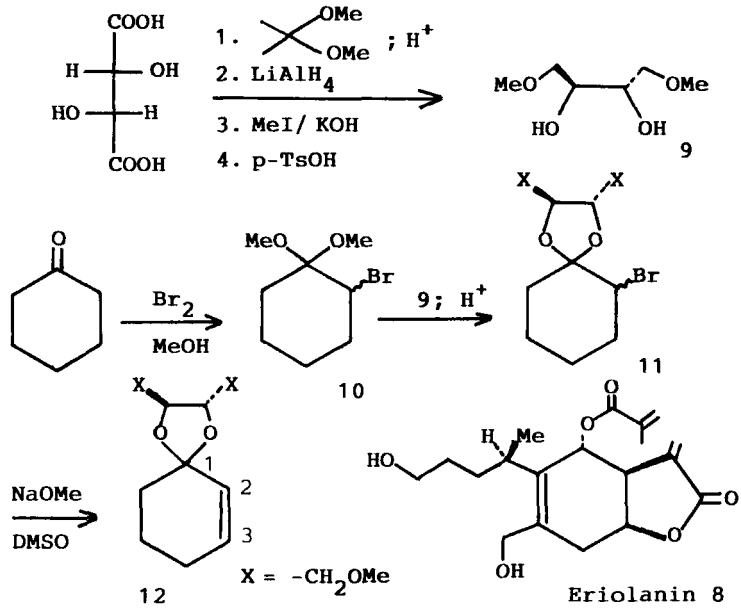
SCHEME 1

In our programme directed towards the synthesis of Eriolanin[6] **8** (scheme 2) and related natural products we considered the possibility of using 2,3-cyclohexene acetal as starting compound and building up the α-methylene-γ-butyrolactone moiety in a regioselective manner. The acetal group is obviously expected to give rise to the hydroxyl function eventually. As chiral acetals have been recognised as excellent auxiliaries[7], it occurred to us to synthesise the butyrolactones which contain chiral acetal unit. Eventual conversion of the bicyclic α-methylene-γ-butyrolactones into natural products would be useful also in

determining the extent of asymmetric induction due to chiral acetal moiety.

The chiral diol employed by us was derived from 2R,3R(+) tartaric acid as reported in the literature[8](scheme 2).Cyclohexanone was first converted[9] into the corresponding α-bromodimethyl acetal **10** which was then exchanged with the diol **9** under acidic conditions to furnish **11**. Elimination of an element of HBr from **11** with NaOMe in DMSO gave the required olefinic acetal **12**.

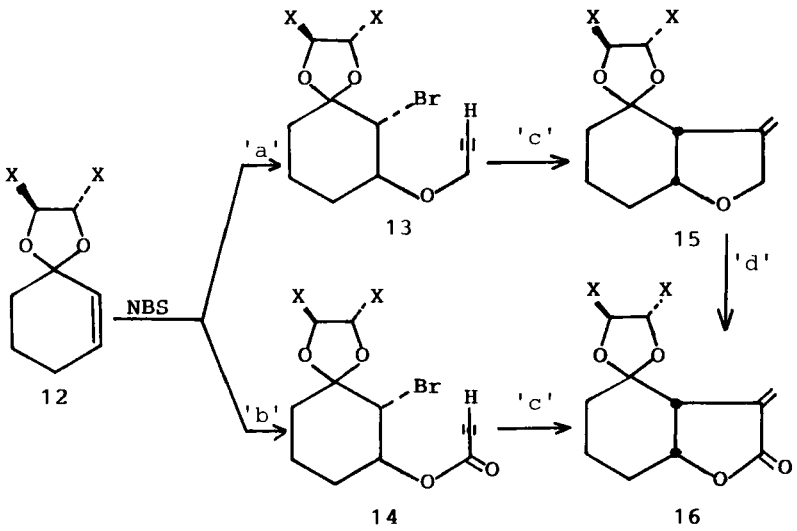
Reactions of olefins with N-bromosuccinimide (NBS) or N-iodosuccinimide followed by the reaction with propargyl alcohol[10] or propiolic acids[11] have been used in the formation of α-methylene-γ-butyrolactones. Since these reactions were performed using radical chemistry the geometry at the ring junction was always cis, an important feature to be noted. The presence of cis α-methylene-γ-butyrolactone unit in Eriolanin and the fact that the reactions of 2,3-olefinic acetals with electrophiles such as NBS or $HgCl_2$ followed by nucleophiles was found to be regioselective, the nucleophile occupying a position farther from the acetal moiety i.e. at C-3 , allowed us to use the radical chemistry approach for their formation. Thus **12** upon sequential treatment with NBS and propargyl alcohol or propiolic acid gave the corresponding addition products **13** and **14** in 96% and 42% yields respectively (scheme 3). Subjection of **13** to n-bu_3SnH treatment in the presence of catalytic amounts of 2,2'-azobisisobutyronitrile (AIBN) in refluxing benzene furnished the ether **15** in 60% yield. Oxidation of **15** with pyridine-chro-



SCHEME 2

mium trioxide gave the ∝-methylene-γ-butyrolactone **16** in 70% yield. Likewise **14** upon treatment with n-bu_3SnH and AIBN directly gave **16** albeit in low yield(34%). The spectral characteristics of **16**, obtained via both these routes were identical.

In order to synthesise more functionalised bicyclic ∝-methylene-γ-butyrolactones we adopted our[2] nitromercuration approach on **12**. Thus **12**,upon treatment with $HgCl_2$/ $NaNO_2$ followed by alkali gave the corresponding nitroolefin **17** in 66% yield (scheme 4). Reaction of **17** with propargyl alcohol in the presence of NaH yielded the addition

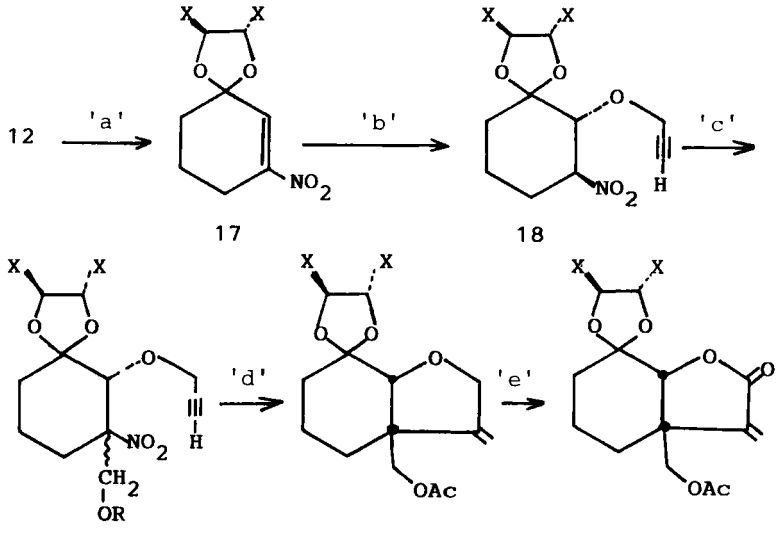


a : $HO-CH_2-C\equiv CH$; b : $HC\equiv C-CO_2H$; c : $n\text{-}bu_3SnH$ / AIBN ;
d : Pyridine-CrO_3 ; X : $-CH_2-OMe$

SCHEME 3

product **18** in 79% yield. Further treatment of **18** with formaldehyde in the presence of aqueous alkali gave **19** whose immediate acetylation[12] (acetic anhydride-pyridine) gave **20** in 82% yield. Compound **20** was subsequently treated with $n\text{-}bu_3SnH$/AIBN for effecting the radical cyclisation to form the exo methylene ether **21** (50%) which gave the α-methylene-γ-butyrolactone **22** upon pyridine-CrO_3 oxidation in 55% yield.

Further conversion of **16** into Eriolanin and of **22** into useful intermediates and ascertaining the extent of asymmetric induction due to chiral acetal is being carried out.



SCHEME 4

Experimental :

General Methods : ^{1}H NMR spectra were recorded on EM 390, Jeol PMX 60 spectrometers with $(CH_3)_4Si$ as internal standard. IR spectra were recorded on Perkin-Elmer 580 and 1320 spectrophotometers by using samples as neat liquids or in $CHCl_3$. Mass spectra were recorded at 70 ev on a Jeol JMS - 3000 D mass spectrometer. Sodium dried THF was distilled from $LiAlH_4$ prior to use.

Preparation of 2-cyclohexene-1-one Cyclic (1S,2S)-1,2-Bis [(methoxy)methyl]ethylene Acetal 12 : To a solution of cyclohexanone (1.96 g, 20 mmol) in 25 ml anhydrous methanol was added a small portion of bromine.The solution was warmed slightly so that the uptake of bromine was complete. The remainder of bromine (3.2 g, 20 mmol) was then added at 35-40°c at such a rate that faint colouration of Br_2 was maintained all the times. After stirring for another 10 min., the reaction mixture was poured into a stirred mixture of NaOMe (2 g) and 20 ml of pet. ether (40-60°c) cooled in an ice-water bath. After stirring for 5 min., 25 ml of cold water was added to it. Usual work up gave **10** which was used as such for furter reaction.

A mixture of **10** (2.24 g, 10 mmol), (-)(2S,3S)-1,4-dimethoxy-2,3-butanediol **9** (1.6g, 10.7 mmol) and anhydrous p-toluene sulphonic acid (52 mg, 0.3 mmol) in dry benzene (10 ml) was refluxed gently for 2.5 hr. After cooling , the reaction mixture was treated with satd. aq. $NaHCO_3$ solution(10 ml) followed by extraction of the product into ether (3 x 20 ml). Further usual work up gave **11** which also was used for the next reaction without purification.

Sodium methoxide (325 mg, 6 mmol) was taken in dry DMSO (5 ml) and stirred at 40°c until a homogeneous mixture was obtained. Compound **11** (1.55 g, 5 mmol) was then slowly added to it at 20°c and then stirred at 50°c for 10 hr. After cooling the reaction mixture was poured onto 10 ml ice-cold water and extracted with ether (4 x 15 ml). Usual work up gave crude **12** which was purified by distillation. Yield:

0.76 g (66%); b.p. 100-105°c/0.1 mm. IR (neat) 3030, 1640 cm^{-1} ^{1}H NMR (CCl_4): δ 1.45-1.87 (4H, m, 2 x CH_2s'), 1.87-2.15 (2H, m, allylic-CH_2-), 3.25-3.6 (10 H, m, 2 x -CH_2-O-CH_3), 3.77-4.03 (2H, m, H-C(O)-C(O)-H), 5.4-5.93 (2H, m,olefinic); mass spectrum m/e: 228. Anal. Calcd. for $C_{12}H_{20}O_4$: C, 63.13; H, 8.83. Found: C,63.0 H,8.68%.

Preparation of 3-propargyloxy-2-bromocyclohexanone cyclic (1S,2S)-1,2-bis[(methoxy)methyl] ethylene acetal 13 : To a solution of **12** (228 mg, 1 mmol) in 2 ml of propargyl alcohol was added NBS (223 mg, 1.25 mmol) in portions at 0°c. The reaction mixture was stirred at 0-10°c for 2 hr. After removal of the excess of propargyl alcohol under vacuum the reaction mixture was quenched with satd. $NaHCO_3$ solution (10 ml) followed by extraction of the product into ether(3 x 20 ml). The usual work up gave a product which was purified by chromatography (SiO_2; eluent: $CHCl_3$). Yield:350 mg (96%). IR (neat): 3280, 2105 cm^{-1}; ^{1}H NMR (CCl_4): δ 1.3-2.03 (6H, m, 3 x -CH_2s'), 2.43 (1H, t, -C≡C-H, J = 2 Hz), 3.33-3.67 (10 H, m, 2 x -CH_2OCH_3), 3.8-4.43 (4H, m, -C(H)O-, -C(H)Br and H-C(O)-C(O)-H), 4.47 (2H, t, -OCH_2-C≡C-H, J = 2 Hz); mass spectrum m/e 362.

Preparation of bicyclic ether 15 : A mixture of **13** (182 mg, 0.5 mmol), freshly distilled n-bu_3SnH (190 mg, 0.65 mmol)and AIBN (25 mg, 0.15 mmol) in 3 ml dry benzene was heated under reflux for 4 hr in a dry nitrogen atmosphere. After cooling benzene was removed at the pump and most of the organotin compounds was removed by filtration through a small pad of tlc grade silica gel(elution with pet. ether). The crude

product was purified by preparative layer chromatography (eluent, benzene:acetone = 85:15) to obtain **15** as a clear oil. Yield: 85 mg (60%). IR(neat): 1650 cm^{-1}; ^1H NMR(CCl_4): δ 1.35-2.0 (6H, m, 3 x $-CH_2-$), 2.5-2.8 (1H, m, $-\underset{|}{CH}-\underset{|}{C}=$), 3.3-3.6 (10 H, m, 2 x $-CH_2OCH_3$), 3.75-4.27 (5 H, m, $-\underset{|}{C}\underline{H}OC\underline{H}_2\underset{|}{C}=$), 4.7-4.9 (1 H, m, olefinic), 5.0-5.23 (1 H, m, olefinic); mass spectrum m/e 284. Anal. Calcd. for $C_{15}H_{24}O_5$: C,63.66; H,8.51. Found: C,63.4; H,8.41%.

Oxidation of 15 to prepare the bicyclic lactone 16 : Chromium trioxide (400 mg, 4 mmol) was added to a solution of dry pyridine (390 mg, 4.9 mmol) in 4 ml of dry CH_2Cl_2 at 15-20°c and the resulting solution was stirred at 25°c for 30 min. A solution of **15** (57 mg, 0.2 mmol) in 1 ml CH_2Cl_2 was then added to it and the whole was refluxed for 1.5 hr.It was cooled and treated with satd. $NaHCO_3$ solution and stirred for 30 min. During this time the solid materials went into the solution. The organic layer was separated and the aqueous layer extracted with ether (3 x 10 ml). The combined organic layers were washed with water and brine and dried over Na_2SO_4. Removal of the solvent followed by purification by preparative chromatography (eluent, benzene: acetone = 80:20) (PLC) gave **16** as thick oil. Yield: 42 mg(70%). IR(neat):3010, 1760 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.4-2.0 (6 H, m, 3 x $-CH_2-$), 3.07 (1H, ddd, $-\underset{|}{CH}\underset{|}{C}=CH_2$, J = 2 Hz, 6 Hz), 3.3-3.6 (10 H,m, 2 x $-CH_2OCH_3$), 3.73-4.07 (2H, m, H-$\underset{|}{\overset{O}{C}}-\underset{|}{\overset{O}{C}}$-H), 4.33-4.7(1H, m, $-\underset{|}{C}\underline{H}O-$),5.67-5.87 (1H, m, olefinic), 6.07 (1H, t, olefinic, J = 2 Hz); mass spectrum m/e 298. Anal. Calcd. for $C_{15}H_{22}O_6$: C,60.39; H,7.43. Found: C,60.51; H,7.5%.

Preparation of 14: A mixture of **12** (228 mg, 1 mmol), propiolic acid(140 mg, 2 mmol) and NBS (232 mg, 1.3 mmol) in 7 ml of dry $CHCl_3$ was refluxed for 8 hr. After cooling the reaction mixture was diluted with 20 ml of $CHCl_3$ and washed with satd. $NaHCO_3$ solution(10 ml), water(10 ml) and brine(10 ml).Evaporation of the solvent after drying(Na_2SO_4) gave a crude product whose purification by column chromatography (SiO_2,eluent, pet.ether:ethyl acetate = 90:10) yielded **14** as a thick oil. Yield: 160 mg (42%). IR(neat): 3250,2120,1718 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.33-2.3 (6H, m, 3 x $-CH_2-$), 2.83 (1H, s, $-C\equiv C-H$),3.3-3.65 (10H, m, 2 x $-\underline{CH_2}O\underline{CH_3}$),3.83-4.2 (3H, m, $-\overset{|}{C}HBr$ & $H-\overset{|}{\underset{O}{\overset{|}{C}}}-\overset{|}{\underset{O}{\overset{|}{C}}}-H$), 4.8-5.2 (1H, m, $-\overset{|}{C}H-O-\underset{O}{\overset{||}{C}}-$); mass spectrum m/e 376. Anal. Calcd. for $C_{15}H_{21}BrO_6$: C,47.76; H,5.61. Found: C,47.66; H,5.7%.

Preparation of the bicyclic lactone 16 from 14 via radical cyclisation : As described for the preparation of **15**, compound **14** (150 mg, 0.4 mmol) was reacted with n-bu_3SnH(150 mg, 0.52 mmol) and AIBN (20 mg, 0.12 mmol) in benzene (4 ml). The product was purified by column chromatography (eluent, pet. ether: ethyl acetate=90:10) to obtain **16**. Yield: 40 mg(34%). Its spectral characteristics were same as described above.

Preaparation of 3-nitro-2-cyclohexene-1-one cyclic (1S,2S)-bis [(methoxy)methyl] ethylene acetal 17 : Mercuric chloride (544 mg, 2 mmol) was added in an aqueous solution(5 ml) of $NaNO_2$(276 mg, 4 mmol). The resulting pale yellow solution was brought in contact with the olefin **12** (456 mg, 2 mmol) and vigorous stirring was maintained at 25°c for 16 hr.Aqueous layer was separated from the viscous nitromercurial by careful decantation and it was extracted with CH_2Cl_2(2 x10 ml).

The combined CH_2Cl_2 layer was used to dissolve the main bulk of the nitro mercurial. It was then treated with a solution of NaOH (80 mg, 2 mmol) in 1 ml of water at -5°c to 0°c and then stirred at room temperature for 20 min. The precipitated mercury was separated by filtration through a pad of celite and the celite cake was washed with CH_2Cl_2 (40 ml). Evaporation of the solvent gave a crude product whose purification by chromatography led to pure **17** as a pale yellow liquid. Yield: 360 mg(66%). IR(neat): 1520 cm^{-1}; ^1H NMR(CCl_4): δ 1.4-2.13 (4H, m, 2 x $-CH_2-$), 2.45-2.8 (2H, m, allylic $-CH_2-$), 3.3-3.7 (10H, m, 2 x $-C\underline{H}_2O\underline{CH}_3$), 3.8-4.1(2H, m, H-C(O)-C(O)-H), 7.0 (1H, br.s, olefinic); mass spectrum m/e 243(M^+-28), 227(M^+-46). Anal.Cal. for $C_{12}H_{19}NO_6$: C,52.74; H,7.01; N,5.13. Found: C,52.82; H,7.11; N,4.98%.

Preparation of 3-nitro-2-propargyloxy-cyclohexanone cyclic (1S,2S)-1,2-bis[(methoxy)methyl]ethylene acetal 18 : To a stirred suspension of NaH(50% dispersion in mineral oil,58mg, 1.2 mmol) in 1 ml of dry THF was added propargyl alcohol(67mg, 1.2 mmol) at 0°c and stirring continued(1 hr.)at 0-10°c till evolution of gas stopped. To this was added a solution of **17** (273 mg, 1 mmol) in 0.5 ml of THF at -10°c and the resulting mixture was stirred at 0-5°c for 2hr. THF was then removed under vacuum and the residue was treated with a soln.of 5g NH_4Cl in 5 ml ice-cold water and then extracted with ether (4 x 15 ml). Usual further work up gave a crude product which was purified by PLC (eluent, benzene:acetone=92:8) to obtain **18** as a thick oil. Yield: 260 mg(79%). IR(neat): 3280,2115, 1545 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.2-2.37(6H, m, 3 x $-CH_2-$), 2.43(1H,

br.t, ≡CH), 3.3-3.8(10H, m, 2 x $-C\underline{H}_2O\underline{CH}_3$), 3.87-4.8(6H, m, $-C\underline{H}OC\underline{H}_2C\equiv$, $-C\underline{H}NO_2$ & ); mass spectrum m/e 283(M^+-46).

Preparation of 3-(acetoxymethyl),3-nitro-2-propargyloxy-cyclohexanone cyclic (1S,2S)-1,2-bis[(methoxy)methyl]ethylene acetal 20 : A mixture of **18** (165 mg, 0.501 mmol),37% formaldehyde (46 mg, 0.6 mmol) and NaOH (3 mg, 0.075 mmol) in 1 ml of isopropanol was stirred at room temperature(15-20°c) for 20 hr. Isopropanol was removed under vacuum and the reaction mixture diluted with 5 ml brine followed by extraction of the product into CH_2Cl_2(4 x 10 ml).Usual work up later gave a product which was immediately acetylated by stirring with acetic anhydride (70 mg, 0.69 mmol) and pyridine (0.5 ml) in 1 ml CH_2Cl_2 at 20°c for 12 hr. After work up in usual manner followed by purification by PLC(eluent, benzene:acetone= 90:10) **20** was obtained as an oil. Yield: 165 mg(82%). IR(neat) 3300, 2115, 1745, 1545 cm^{-1}; ^1H NMR ($CDCl_3$):δ1.4-2.6(10H, m, ≡CH, 3 x $C\underline{H}_2$- and a singlet at 2.03 due to $-O\overset{O}{\overset{\|}{C}}-CH_3$),3.3-3.8 (10H, m, 2 x $-C\underline{H}_2O\underline{CH}_3$), 3.9-4.9 (7H, m, $-C\underline{H}_2OAc$, $-C\underline{H}OC\underline{H}_2C\equiv$ and ); mass spectrum m/e 355 (M^+-NO_2).

Denitrative cyclisation of 20 to bicyclic ether 21 :A mixture of **20** (160 mg, 0.4 mmol), freshly distilled n-bu_3SnH (150 mg,0.52 mmol) and AIBN (20 mg, 0.12 mmol) in benzene(3ml) was refluxed for 4 hr under nitrogen atmosphere. Benzene was then removed under reduced pressure and then the residue was washed with pet.ether to remove the organotin compounds. Purification by PLC (eluent , benzene:acetone=90:10, double elution) gave a thick oil.Yield:71mg(50%). IR(neat):3020, 1740 cm^{-1}; ^1H NMR($CDCl_3$): δ1.4-2.03(6H, m, 3 x $-CH_2$-),2.05(3H,

s, -OCCH$_3$), 3.3-3.75(10H, m, 2 x -CH$_2$OCH$_3$), 3.8-4.3(5H, m, -CHO-, -CH$_2$OAc and H-C(O)-C(O)-H), 4.4-4.7(2H, br.s, -OCH$_2$-C=), 4.93(2H,br. s, =CH$_2$); mass spectrum m/e 356. Anal. Calcd. for $C_{18}H_{28}O_7$: C,60.66; H,7.92. Found: C,60.56; H,7.81%.

Preparation of the bicyclic α-methylene-γ-butyrolactone 22 : via oxidation of 21 : A complex from chromium trioxide(400 mg, 4 mmol) and pyridine (390 mg, 4.9 mmol) was prepared as described above (cf. oxidation of **15**). To this was then added a solution of **21** (70 mg, 0.197 mmol) in 1 ml CH_2Cl_2 and refluxed for 1.5 hr. It was cooled, treated with satd. $NaHCO_3$ solution(5ml) and stirred for 30 min.The organic layer was separated and the aqueous layer extracted with ether(3 x 10 ml). Organic layers were combined and further worked up as usual to get a crude product whose purification by PLC(double elution, benzen:acetone=90:10) yielded **22** as a thick oil.Yield:40 mg (55%).IR (neat)1770, 1735 cm^{-1}; ^{1}H NMR($CDCl_3$): δ1.35-2.0(6H, m, 3 x -CH$_2$-), 2.05 (3H, s, -O-C(O)-CH$_3$), 3.3-3.7(10H, m, 2 x -CH$_2$-OCH$_3$), 3.8-4.27(4H, m, H-C(O)-C(O)-H and -CH$_2$OAc), 4.37(1H, s, -CH-O-C(O)-), 5.45 (1H, d, olefinic, J = 2.5 Hz), 6.25 (1H, d,olefinic, J = 2.5 Hz); mass spectrum m/e 370. Anal.Calcd. for $C_{18}H_{26}O_8$: C,58.37; H,7.08. Found: C,58.58;H,7.2%.

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