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AN EFFICIENT SYNTHESIS OF THE PRELOG-DJERASSI LACTONE METHYL ESTER FROM (-)-*TRANS*-PULEGENIC ACID

Salih Hacini [a] and Maurice Santelli *[b]

[a]*Laboratoire de Synthèse Organique, Université d'Oran-Es Sénia (Algeria)*
[b]*Laboratoire de Synthèse Organique associé au CNRS n° 1411, Centre de St-Jérôme, Av. Esc. Normandie-Niemen13397 Marseille Cedex 13 (France).*

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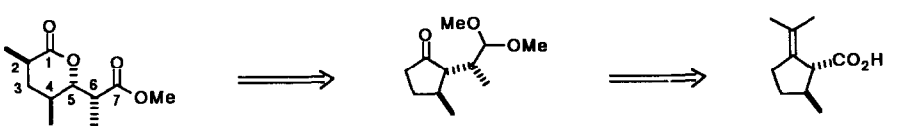
Abstract : The (+)-Prelog-Djerassi lactone methyl ester 1 was obtained from trans-pulegenic acid 2 in seven steps in 22 % overall yield; three more steps from an unwanted diastereoisomer gave additionnal 5 %.

Résumé : L'ester méthylique de la lactone de Prelog-Djerassi 1 a été obtenu en sept étapes à partir de l'acide pulégénique 2 avec un rendement global de 22 %; le traitement en trois étapes d'un diastéréoisomère non souhaité fournit 5 % supplémentaire.

The Prelog-Djerassi lactone is a degradation product of methymycin (1) and narbomycin (2) and a synthon for the preparation of several macrolide antibiotics. From 1975 (3), considerable attention has been given to the synthesis of the Prelog-Djerassi lactone derivatives, but only few works were devoted to the obtention of (+)-Prelog-Djerassi lactone methyl ester 1 (4,5).

We recently explored a new synthetic strategy which involves the use of pulegenic acid 2 in the obtention of the Grieco intermediate (6,7). We present here, an efficient synthesis of the Prelog-Djerassi lactone ester 1. Retrosynthetic analysis identified (*S*)-pulegenic acid 2 as a logical precursor. The main feature was the elaboration of the exocyclic functionality.



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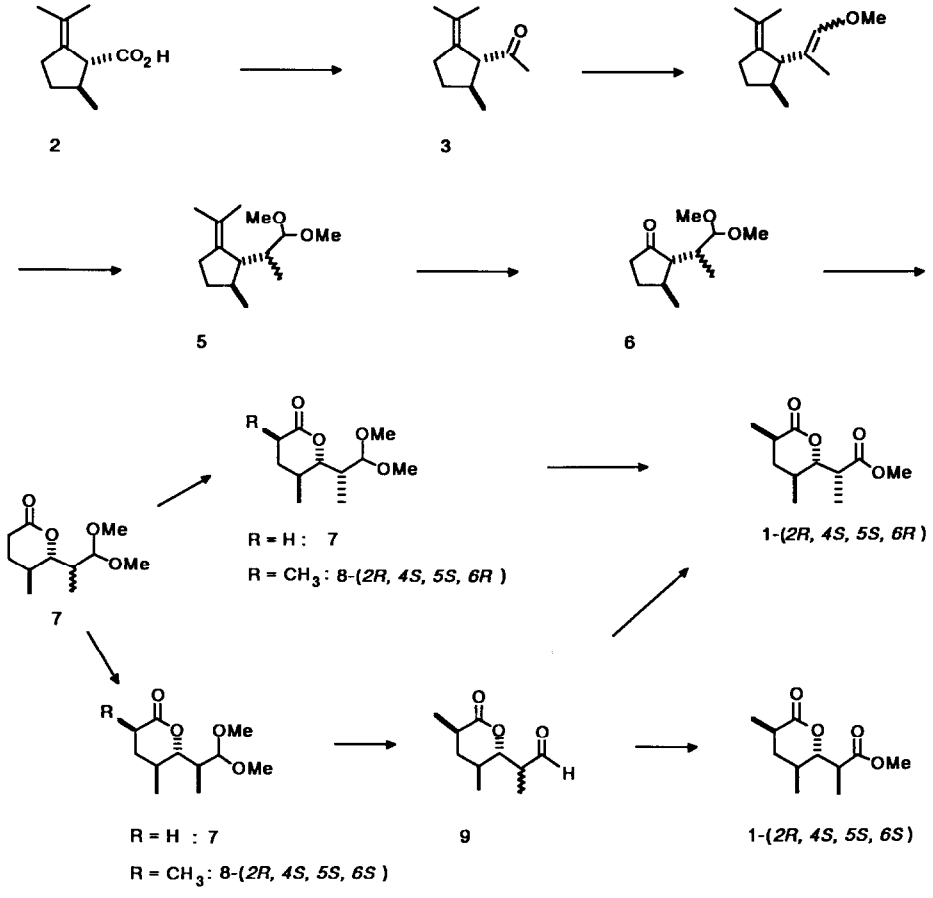
(2*R*, 6*S*)-Pulegenic acid was obtained from (*S*)-pulegone in good yield (67-70 %). A C2-homologation was performed in two steps : methyllithium addition giving the acetylcyclopentane 3 (92 % yield) and Wittig reaction with (methoxymethyl)triphenylphosphonium ylide leading to a *E*/*Z* mixture (4/1) of the enol ether 4

(7)(95 % yield). Hydrolysis afforded an epimeric mixture of aldehydes. However, addition of methanol furnished the corresponding dimethylacetals 5 (95 % yield) with the right isomer 5-(*R*) as major product (5-(*R*)/5-(*S*) : 7/3)(8). The mixture of 5, difficult to purify by flash chromatography, successively underwent ozonolysis (90 % yield) and m-chloroperbenzoic acid (9) treatment leading to the lactones 7 (91 % yield). After chromatographic separation, the 7-(6*R*) and 7-(6*S*) isomers (respectively 7/3) were methylated (87 % yield)(10,11). The expected 1/1 epimeric pair of lactones 8 was equilibrated with *tert*-BuOK/*tert*-BuOH (10d) giving 8-(2*R*)(12) as the main isomer (8-(2*R*)/8-(2*S*) : 6/1).



Ozonolysis of 8-(2*R*, 6 *R*) isomer according to the Deslongchamps procedure (13) led to the (+)-Prelog-Djerassi lactone methyl ester 1-(2*R*, 4 *S*, 5*S*, 6 *R*)(70 % yield). The unwanted isomer 8-(2*R*, 6*S*) resulting from the methylation and equilibration of 7-(6*S*) was hydrolyzed in aldehyde 9 (14). An equilibration reaction occurred in favour of the desired isomer 6 *R* (9-(6*R*)/9-(6*S*) : 3/2). Oxidation in acid **10** with the Jones reagent (15) and esterification with diazomethane (69 % yield) gave a mixture of 1-(2*R*, 4*S*, 5*S*, 6*R*) and 1-(2*R*, 4*S*, 5*S*, 6*S*)(3/2) (16) which was separated.

Conclusion : The Prelog-Djerassi lactone methyl ester 1-(2*R*, 4*S*, 5 *S*, 6*R*) was prepared in 22 % yield starting from pulegenic acid. Using the unwanted isomer 7-(6*S*), the overall yield of this synthesis rose to 27 %.

Experimental Section

General Methods. ^1H NMR and ^{13}C NMR spectra of $CDCl_3$ solutions were determined on Bruker AC 200 and Varian XL 200 (50.309 MHz) spectrometers, respectively. For ^{13}C NMR spectra, assignments were confirmed by *J*-modulated spin echo. Mass spectra were obtained on a Varian MAT 311 spectrometer. All reactions were carried out under a positive argon atmosphere. The glassware was dried at 160°C, assembled hot, and cooled in a dessicator with nitrogen atmosphere. Flash column chromatography used Merck grade silica gel (230-400 mesh) and t.l.c. Merck 60 F254 silica plates.

Materials. Ether and THF were distilled from ethylmagnesium bromide solutions ; diisopropylamine, *tert*-butanol, pentane were distilled from calcium hydride. Dichloromethane was distilled on P_2O_5.

(2 *S*, 3 *S*)-2-((2 *RS*)-1,1-Dimethoxypropan-2-yl)-3-methyl-1-isopropylidene cyclopentane 5. To a solution of (2 *R*, 3 *S*)-3-methyl-2-(1-methoxy-propen-2 yl)-1-isopropylidene cyclopentane **4** (7)(2.91 g, 15 mmol) in anhydrous methanol (12 mL) was added 3 drops of concentrated hydrochloric acid. The solution was stirred for about 15 h, and as soon as **4** disappeared (t.l.c.), a sodium methylate solution in methanol was added to reach neutrality. After *vacuum* elimination of excess methanol, addition of ether, the solution was washed with water and dried on $MgSO_4$. Concentration *in vacuo* afforded crude acetal **5** used in the next step (3.22 g, 14.2 mmol, 95 %). An analytical sample obtained by extensive flash chromatography (ether-pentane 5:95) was a mixture of *R*/*S* isomers (7/3) at the exocyclic asymmetric center (t.l.c. ether-pentane 3/97, *S*-isomer : R_f = 0.27 ; *R*-isomer : R_f = 0.22). IR (film) 1100-1050, 970-745 cm^{-1} ; mass spectrum *m/e* 226 (0.15), 194 (15) (M - CH_3OH (HRMS calcd for $C_{13}H_{22}O$ 194.1670, found 194.1660), 162 (6), 147 (7), 123 (27), 75 (100). The *R* epimer showed : ^1H NMR δ 4.16 (1, d *J* = 4.8 Hz), 3.36 (6, s), 1.68 (6, br. s), 0.88 (3, d *J* = 6.8 Hz), 0.86 (3, d *J* = 7.0 Hz) ; ^{13}C NMR (in part) δ 137.5 (s), 123.5 (s), 108.1 (d), 55.1 (q), 54.3 (q), 51.1 (d), 39.9 (d), 37.2 (d), 31.4 (t), 28.7 (t), 12.2 (q). The *S* epimer showed ; ^1H NMR δ 4.13 (1, d *J* = 7.4 Hz), 3.34 (6, s), 1.66 (6, s), 0.94 (3, d *J* = 6.8 Hz), 0.80 (3, d *J* = 7.0 Hz) ; ^{13}C NMR (in part) δ 139.0 (s), 122.2 (s), 107.3 (d), 53.6 (q), 52.9 (q), 49.7 (d), 38.2 (d), 34.1 (d), 33.4 (t), 30.2 (t), 11.0 (q).

(2*S*, 3 *S*)-2-((2*RS*)-1,1-Dimethoxypropan-2-yl)-3-methylcyclopentanone 6. Ozone in oxygen was bubbled through a solution of **5** (3.2 g, 14 mmol) in 140 mL of CH_2Cl_2-MeOH (2/1) which contained 2 drops of pyridine and 1 mL of an ethanolic solution of "Sudan III" (Eastman Kodak) (1/10 000 (17)) at -80°C until the solution turned yellow. While the solution was still at -80°C, the system was flushed with nitrogen. The solution was then allowed to warm up to 0°C ; 28 mL of dimethyl sulfide were added and the mixture stirred for 1 h. Washing with brine (2 x 200 mL), extraction of the aqueous layer with CH_2Cl_2, drying ($MgSO_4$) and concentration *in vacuo* gave the crude product **6**. Flash chromatographic purification (ether-pentane 1/3) furnished a mixture of *R*/*S* isomers (2.50 g, 12.9 mmol, 90%) (t.l.c. ether-pentane 1/3, *S*-isomer : R_f = 0.3 ; *R*-isomer; R_f = 0.26). IR (film) 1745, 1110, 1060, 975, 950 cm^{-1} ; mass spectrum *m/e* 200 (0.1), 169 (5) (M - OCH_3) (HRMS calcd for $C_{10}H_{17}O_2$ 169.1228, found 169.1223), 168 (6), 112 (11), 102 (13), 75 (100). The *R* epimer showed : ^1H NMR δ 4.67 (1, d *J* = 8.7 Hz), 3.35 (3, s), 3.31 (3,s), 1.15 (3, d *J* = 6.1 Hz), 0.91 (3, d *J* = 7.0 Hz) ; ^{13}CMR δ 217.7 (s), 104.9 (d), 56.5 (d), 52.4 (q), 51.8 (q), 38.0 (t), 33.7 (d), 33.1 (d), 29.3 (t),

18.9 (q), 11.2 (q). The *S* epimer showed : ^{1}H NMR δ 4.33 (1, d *J* = 8.7 Hz), 3.33 (3, s), 3.29 (3, s), 1.15 (3, d *J* = 6.1 Hz), 0.86 (3, d *J* = 7.0 Hz) ; ^{13}C NMR δ 218.4 (s), 105.4 (d), 56.7 (d), 52.1 (q), 51.3 (q), 37.7 (t), 33.5 (d), 32.8 (d), 29.2 (t), 19.7 (q), 12.0 (q).

(4*S*, 5*S*, 6*R*)-7,7-Dimethoxy-4,6-dimethylheptan-5-olide 7. To a solution of keto-acetal 6 (2.48 g, 12.4 mmol) in CH_2Cl_2 (160 mL) at 0°C, was added $NaHCO_3$ (2.6 g, 31 mmol) and slowly *m*-chloroperbenzoic acid (6.10 g, 35.3 mmol). The reaction mixture was allowed to warm to room temperature and stirred for 36 h (with protection against light). After filtration, the solution was washed with a 10 % solution of $Na_2S_2O_3$, a 5 % solution of $NaHCO_3$, brine and dried on $MgSO_4$. Concentration *in vacuo* and flash chromatographic purification on silica gel (ether-pentane 1/2) afforded 7-(6*R*) (1.78 g, 7.9 mmol, 64%) and 7-(6*S*) (0.74 g, 3.4 mmol, 27%) (t.l.c. ether-pentane 2/1, 6*S* : R_f = 0.29 ; 6*R* : R_f = 0.19). IR (film) 1740, 1255, 1200, 1100, 1005 cm^{-1} ; mass spectrum *m/e* 216 (0.1), 185 (6) (M - OCH_3) (HRMS calcd for $C_{10}H_{17}O_3$ 185.1177, found 185.1172), 113 (15), 85 (9), 75 (100). The 6*R* epimer showed : ^{1}H NMR δ 4.44 (1, d *J* = 8.8 Hz), 4.27 (1, dd *J* = 10,3 Jz and 1.6 Hz), 3.41 (3, s), 3.38 (3, s), 0.99 (3, d, *J* = 7.0 Hz), 0.95 (3, d *J* = 7.0 Hz) ; ^{13}C NMR δ 171.6 (s), 105.4 (d), 84.8 (d), 54.6 (q), 53.5 (q), 37.6 (d), 29.8 (d), 29.8 (t), 28.1 (t), 16.8 (q), 7.9 (q). The 6*S* epimer showed : ^{1}H NMR δ 4.42 (1, d *J* = 7.1 Hz), 4.00 (1, dd *J* = 9.0 Hz and 3.1 Hz), 3.43 (3, s), 3.40 (3, s), 1.07 (3, d *J* = 7.0 Hz), 1.04 (3, d *J* = 6.6 Hz) ; ^{13}C NMR δ 171.6 (s), 105.6 (d), 88.7 (d), 54.8 (q), 53.6 (q), 38.8 (d), 30.1 (d), 29.3 (t), 27.4 (t), 17.7 (q), 13.2 (q).

(2*R*, 4*S*, 5*S*, 6*R*)-7,7-Dimethoxy-2,4,6-trimethylheptan-5-olide 8. To a solution of anhydrous diisopropylamine (610 mg, 6 mmol) in anhydrous THF (10 mL) at -20°C, was added butyllithium (1.5 M in hexane, 4 mL, 6 mmol). The resulting mixture was allowed to warm to room temperature and after 15 min of stirring, cooled at -80°C. A solution of 7-(6*R*) (1.08 g, 5 mmol) in anhydrous THF (8 mL) was slowly added (1 h). After 0.3 h of stirring, a solution of iodomethane (3.11 mL, 50 mmol), HMPA (1.05 mL, 6 mmol) in anhydrous THF (4 mL) was added. After stirring at -80°C for 1 h and at -45°C for 0.75 h, a solution of hydrochloric acid (2N, 5 mL) was added. The solution was allowed to warm to room temperature and extracted with ether. The organic solution was washed with brine, dried ($MgSO_4$) and concentrated *in vacuo*. The crude product quickly purified by flash chromatography on silica gel afforded a mixture of 8-(2*R*) and 8-(2*S*) (1/1) (1.0 g, 4.3 mmol, 87 %).

To a solution of **8** (55.2 mg, 0.24 mmol) in anhydrous *tert*-butanol (24 mL) was added freshly sublimated potassium *tert*-butoxyde (27.3 mg, 0.24 mmol). The reaction mixture was stirred at room temperature for 16 h. A saturated solution of ammonium chloride (5 mL) was added. Usual work-up and extensive flash chromatographic purification on silica gel (ether-pentane 1/2) afforded 8-(2*R*, 6*R*)(12) and 8-(2*S*, 6*R*) (*circa* 6/1) (t.l.c. ether-pentane 2/1, 8-(2*R*, 6*R*) : R_f = 0.29 ; 8-(2*S*, 6*R*) : R_f = 0.32). IR (film) 1740, 1190, 1100-1050, 995 cm^{-1} ; mass spectrum *m/e* 230 (1) (HRMS calcd for $C_{12}H_{22}O_4$ 230.1518, found 230.1523), 199 (2), 198 (2), 127 (14), 75 (100). The 2*R*, 6*R* isomer showed : ^{1}H NMR δ 4.44 (1, d *J* = 8.7 Hz), 4.26 (1, d *J* = 10 Hz), 3.41 (3, s), 3.38 (3, s), 2.48 (1, sext. d *J* = 6.4 Hz, 1.0 Hz), 1.28 (3, d *J* = 7.1 Hz), 0.96 (3, d *J* = 6.4 Hz), 0.94 (3, d *J* = 6.8 Hz) ; ^{13}C NMR δ 174.5 (s), 105.4 (d), 86.0 (d), 54.7 (q), 53.6 (q), 37.8 (d), 37.6 (t), 36.3 (d), 30.6 (d), 17.3 (q), 16.8 (q), 8.0 (q). The 2*S*, 6*R* isomer showed : ^{1}H NMR δ 4.41 (1, d *J* = 8.7 Hz), 4.26 (1, dd *J* = 10.5 and 1.8 Hz), 3.38(3, s), 3.37 (3, s), 2.69 (1, sext. d *J* = 7.0 Hz, 1.1 Hz), 1.22 (3,d *J* = 6.8 Hz), 0.975 (3, d*J* = 6.7 Hz), 0.97 (3, d *J* = 6.9 Hz) ; ^{13}C NMR δ 176.4 (s), 105.5 (d), 82.1 (d), 54.5 (q), 53.6 (q), 37.2 (d), 35.2 (t), 32.5 (d), 28.2 (d), 17.3 (q), 16.5 (q), 7.9 (q).

(+)-Prelog-Djerassi lactone methyl ester 1-(2*R*, 4*S*, 5*S*, 6*R*). Ozone in oxygen was bubbled through a solution of **8**-(2*R*,6*R*)(161 mg, 0.7 mmol) in ethyl acetate (12 mL) at 10 °C for 8 h (t.l.c.), then the system was flushed with nitrogen. Concentration *in vacuo*, addition of ether, washing with Na_2CO_3 (5 %), brine and flash chromatographic purification afforded Prelog-Djerassi lactone methyl ester (105 mg, 0.49 mmol, 70 %) : IR (film) 1745, 1190, 1100, 1000 cm^{-1}; mass spectrum *m/e* 214 (1)(HRMS calcd for $C_{11}H_{18}O_4$ 214.1205, found 214.1208), 183 (4), 144 (16), 127 (45), 113 (14), 99 (17), 43 (100); ^1H NMR δ 4,54 (1, dd *J* = 10.2 and 2.5 Hz), 3.70 (3, s), 1.27 (3, d *J* = 7.1 Hz), 1.21 (3, d *J* = 7.0 Hz), 1.02 (3, d *J* = 6.8 Hz); ^{13}C NMR δ 175.5 (s), 173.7 (s), 86.3 (d), 52.2 (q), 41.3 (d), 37.4 (t), 36.3 (d), 31.0 (d), 17.3 (q), 17.0 (q), 8.7 (q). From the isomer **8**-(2*S*, 6*R*), the same procedure led to **1**-(2*S*, 4*S*, 5*S*, 6*R*) : ^{13}C NMR δ 175.5 (s), 173.5 (s), 82.8 (d), 52.2 (q), 41.1 (d), 34.9 (t), 32.5 (d), 28.8 (d), 17.4 (q), 16.4 (q), 9.2 (q).

Obtention of (+)-Prelog-Djerassi lactone methyl ester 1 from 7-(4*S*, 5*S*, 6*S*). The above procedure for the methylation and equilibration were repeated with **7**-(4*S*, 5*S*, 6*S*). **8**-(2*R*, 4*S*, 5*S*, 6*S*) and **8**-(2*S*, 4*S*, 5*S*, 6*S*) (*c.a* 6/1) were separated by flash chromatography on silica gel (t.l.c. ether-pentane 2/1, respectively : R_f = 0.34 and 0.37). The 2*R* epimer showed : ^1H NMR δ 4,46 (1, d *J* = 7.0 Hz), 3.95 (1, dd *J* = 9.5 and 2.3 Hz), 3.35 (6, br. s), 1.21 (3, d *J* = 7.0 Hz), 1.07 (3, d *J* = 7.0 Hz), 1.04 (3, d *J* = 7.0 Hz), 1.04 (3, d *J* = 6.7 Hz) ; ^{13}C NMR δ 174.7 (s), 105.6 (d), 90.1 (d), 54.6 (q), 53.1 (q), 38.3 (d), 37.6 (t), 36.1 (d), 31.7 (d), 17.9 (q), 17.2 (q), 14.0 (q). The 2*S* epimer showed : ^1H NMR δ 4.49 (1, d *J* = 7.0 Hz), 3.98 (1, dd *J* = 9.0 and 3.8 Hz), 3.37 (6, br. s), 1.23 (3, d *J* = 6.9 Hz), 1.08 (3, *J* = 6.9 Hz), 1.03 (3, d *J* = 6.8 Hz) ; ^{13}C NMR δ 176.1 (s), 105.4 (d), 86.4 (d), 54.9 (q), 53.7 (q), 38.7 (d), 35.0 (t), 32.3 (d), 28.3 (d), 18.2 (q), 16.6 (q), 12.6 (q).

To a solution of **8**-(2*R*, 4*S*, 5*S*, 6*S*) (230 mg, 1 mmol) in THF (4 mL) was added hydrochloric acid (0.5 N, 2 mL). The solution was stirred for about 20 h (t.l.c.). After addition of $NaHCO_3$ and usual work-up, crude aldehyde **9** was obtained. To a solution of crude **9** (123 mg, 0.66 mmol) in acetone (8 mL) at -12°C, was added Jones reagent (0.41 mL, from CrO_3 (2.67 g), conc. H_2SO_4 (2.3 mL) and water to obtain 10 mL). The mixture was stirred for 1 h, 2-propanol was added, filtered and concentrated *in vacuo*. The residue was diluted with acidic water (pH 1), extracted with ether, washed with brine, dried ($MgSO_4$) and filtered. To the solution, excess ethereal solution of diazomethane was added. The solution was concentrated *in vacuo*. Extensive flash chromatographic purification afforded Prelog-Djerassi lactone methyl ester **1** (*c.a* 90 mg, 0.42 mmol, 42%) and **1**-(2*R*, 4*S*, 5*S*, 6*S*) (*c.a* 60 mg, 0.28 mmol, 28%) : ^1H NMR δ 4.19 (1, dd *J* = 10.2 and 2.7 Hz), 3.70 (3, s), 1.32 (3, d *J* = 7.1 Hz), 1.27 (3, d *J* = 7.2 Hz), 0.99 (3, d *J* = 6.0 Hz) ; ^{13}C NMR δ 175.5 (s), 173.6 (s), 87.6 (d), 51.8 (q), 42.1 (d), 37.4 (t), 36.1 (d), 31.3 (d), 17.1 (q), 16.8 (q), 12.8 (q).

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