# Total synthesis of *d,l*-coronafacic acid by an intermolecular Diels–Alder approach

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An efficient total synthesis of *d,l*-coronafacic acid (**2**) has been achieved from 4-cyclopentene-1,3-dione (**4**). The synthesis involves keto ester **7** as a key intermediate that is conveniently prepared by the Diels–Alder addition of enedione **4** to ethyl 4-ethyl-2,4-pentadienoate (**5**).

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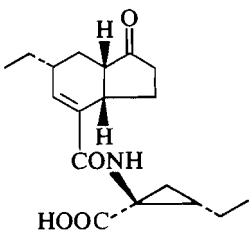
Utilisant la cyclopentène-4 dione-1,3 (**4**) comme produit de départ, on a réalisé une synthèse totale efficace de l'acide (*d,l*)-coronafacique (**2**). Le céto-ester **7**, l'intermédiaire clé dans cette synthèse, se prépare facilement par une addition de Diels–Alder de l'ènedione **4** sur l'éthyl-4 pentadiène-2,4 oate d'éthyle (**5**).

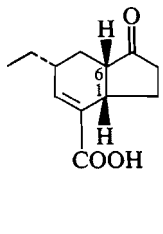
[Traduit par le journal]



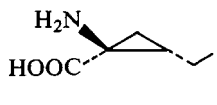
In 1977, Ichihara *et al.* (1) reported the isolation, from culture broth of *Pseudomonas coronafacience var. atropurpurea*, of a phytotoxic compound (2) named coronatine. These investigators were also able to rigorously establish the structure (1) and the absolute stereochemistry (3, 4) of the dextrorotatory phytotoxin as **1**. Hydrolysis of coronatine (**1**) gave (+)-coronafacic acid (**2**) and (+)-coronamic acid (**3**). Conversely, condensation of coronafacic acid (**2**) via the corresponding acid chloride and coronamic acid (**3**) gave rise to the parent molecule **1** (3). Coronafacic acid (**2**), which was found to be also present in the culture broth of the phytopathogenic bacterium (1), has been the subject of considerable synthetic activity and several total syntheses have been accomplished during the past few years (5). We wish to describe an efficient total synthesis of the racemic coronafacic acid (**2**) based on a fundamentally different approach.




**1**




**2**




**3**

As illustrated in Scheme 1, the synthesis began with a Diels–Alder reaction whereby the complete carbon framework of the target molecule was conveniently assembled. 4-Cyclopentene-1,3-dione (**4**) was chosen as the dienophile for its high dienophilicity and for its symmetry, as the use of unsymmetrical 2-cyclopentenones is expected to produce adducts such as **6** possessing the undesirable regiochemistry according to the *ortho* and *para* rules (6) governing the Diels–Alder reaction. In refluxing toluene, the cycloaddition of enedione **4** and ethyl 4-ethyl-2,4-pentadienoate (**5**) (7) proceeded smoothly to give a 67% yield of the crystalline adduct **7**,[1] mp 86–88°C. The nmr spectrum showed that the compound was completely enolized in chloroform solution and that the two stable enols were in a ratio of ca. 5:1.

Treatment of the lithium salt of diketone **7**, generated *in situ* using lithium hydride as a base, with phenyl dichlorophosphate and lithium chloride in tetrahydrofuran in the presence of a small amount of hexamethylphosphoramide[2] at room temperature overnight resulted in the formation of two isomeric chlorides **8** and **9** in 3:2 ratio and in a total yield of 64%. The two isomers were separated by high pressure liquid chromatography and their regiochemistry was individually assigned on the basis of the following observations. In the nmr spectrum, the C-1 proton of the major isomer appeared at δ 3.51 as a doublet of doublets of doublets of doublets with coupling constants of 7, 7, 2.5, and 2 Hz. This proton was shown by spin decoupling experiments to be coupled to the enone vinyl proton which appeared at δ 6.21 as a doublet with a small coupling constant of 2 Hz. On the other hand, the enone vinyl proton (doublet at δ 6.23) of the minor isomer was found to be coupled with a coupling constant of 2 Hz to the C-1 proton at δ 3.78 (ddd, $J = 7$, $J' = J'' = 2$ Hz).

The isomeric chlorides **8** and **9** were individually transformed to the desired keto ester **10** as follows. The conversion of **9** into **10** was straightforward; on hydrogenation in benzene in the presence of 5% palladium on carbon and sodium bicarbonate,[3] the former compound underwent selective reduction to give an 83% yield of keto ester **10** as a mixture of two stereoisomers (~9:1). For the transformation of **8** → **10**, initial studies were intended to proceed via the intermediacy of alcohol **11** that could be easily prepared by the reduction of **8** with sodium borohydride in ethanol. Unfortunately, alcohol **11** underwent extensive decomposition when treated with a variety of acids and with titanium tetrachloride and failed to give any detectable amount of the desired product **16**. To circumvent this difficulty, chloride **8** was subjected to the treatment with silver nitrate in hot methanol[4] and the resulting esters **12** and **13**, obtained in 82% yield and in ca. 9:1 ratio, were reduced with lithium aluminum hydride.[5] Acidic work-up using hydro-

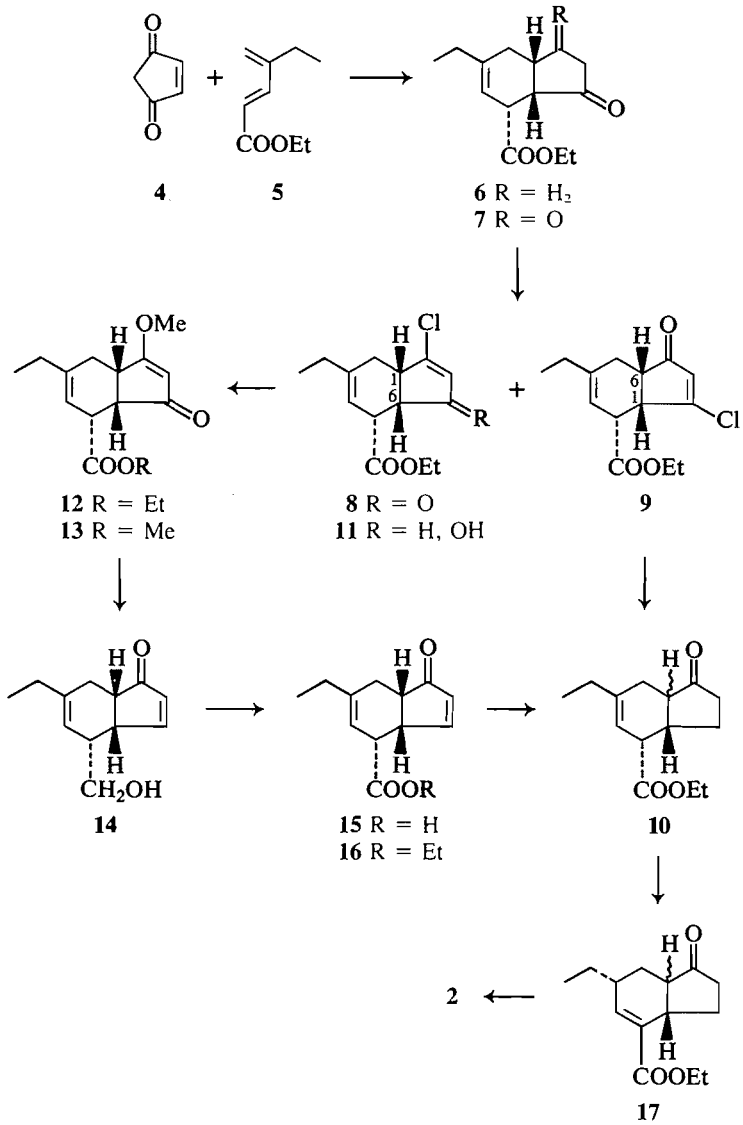
[1]The assignment of its stereochemistry follows from the *endo*-rule and the *cis*-principle (6).

[2]This procedure was developed after several unsuccessful attempts were made to effect the transformation using known methods (8). We are currently examining the generality of this procedure for the conversion of β-dicarbonyl compounds to β-halo-α,β-unsaturated carbonyl compounds. Results will be reported elsewhere.

[3]When the catalytic hydrogenation reaction was carried out in the absence of sodium bicarbonate, a substantial amount of the fully saturated compound (dihydro derivative of **10**) was formed.

[4]When performed in ethanol, the reaction was very slow and gave a poor yield of ester **12**.

[5]Selective reduction of the enone carbonyl with sodium borohydride and with lithium tri-*tert*-butoxyaluminum hydride was attempted without much success.




SCHEME 1

chloric acid gave a 70% yield of alcohol **14**. Jones oxidation of **14** followed by esterification of the resulting acid **15** with potassium carbonate and ethyl iodide in refluxing acetone (9) gave rise to enone ester **16** in 65% yield. Subsequent hydrogenation of this compound, carried out in ethyl acetate using 5% Pd/C as a catalyst, resulted in the selective saturation of the less substituted carbon–carbon double bond to furnish, in 92% yield, the epimeric keto esters **10** identical with those obtained previously from the minor chloride **9** (*vide supra*). Thus, by a total of seven operations, diketone **7** was converted to **10** in an overall yield of 35%.

To effect the isomerization of the double bond, the mixture of epimeric keto esters **10** was subjected to treatment with sodium ethoxide in ethanol at room temperature. Hydrolysis of the resulting α,β-unsaturated esters **17** (71% yield) in refluxing aqueous hydrochloric acid gave, in 73% yield, a solid material consisting of coronafacic acid (**2**) and its C-6 epimer[6] in ca. 4:1 ratio as determined by nmr analysis. Recrystallization of this solid material from ether–hexane gave pure *d,l*-coronafacic acid (**2**) (mp 121–125°C) identical in all respects with an authentic sample.

## Experimental

*General*

Melting points were determined on a Kofler hot stage apparatus and are uncorrected. Elemental analyses were performed by the Microanalytical Laboratory of this department. Infrared spectra (ir) were recorded on a Nicolet 7-199 FT-IR spectrophotometer. Nuclear magnetic resonance spectra (nmr) were recorded on a Bruker WH-200 or WH-400 spectrometer and were obtained on solutions in deuteriochloroform with tetramethylsilane as internal reference. Mass spectra (ms) were recorded using A.E.I. model MS12 or MS50 mass spectrometers. Anhydrous magnesium sulfate was used for drying organic solutions.

*2-Carbethoxy-4-ethylbicyclo[4.3.0]non-3-ene-7,9-dione (7)*

A solution of 4-cyclopentene-1,3-dione (**4**) (290 mg, 3 mmol) and ethyl 4-ethyl-2,4-pentadienoate (**5**) (1.175 g, 7.6 mmol) in toluene (20 mL) was refluxed for 30 h under an atmosphere of nitrogen. The resulting solution was concentrated under reduced pressure. The residue was chromatographed on silica gel. Elution with a solution of 10%

[6]This compound was also isolated directly from the culture broth of *Pseudomonas coronafacience var. atropurpurea* (1). Its epimerization to **2** during the recrystallization of **2** and vice versa have been observed previously (1).

petroleum ether in ethyl acetate gave diketone **7** (500 mg; 67% yield): mp 86–88°C (ether – petroleum ether); ms $M^+$ 250.1202 (calcd. for $C_{14}H_{18}O_4$: 250.1205). The compound existed completely in two enol forms (~5:1) in chloroform solution as indicated by the following spectral data: ir ($CHCl_3$ cast): 3440 (OH), 1730 (ester), 1642, and 1549 (β-hydroxy-α,β-unsaturated ketone) $cm^{-1}$; nmr: two sets of signals with the major at δ: 6.04 (br s, 1H, —OH), 5.58 (dd, 1H, $J$ = 6, $J'$ = 1 Hz, =CH—), 5.26 (s, 1H, =CHCO—), 4.24 (q, 2H, $J$ = 7 Hz, —$OCH_2$—), 2.06 (q, 2H, $J$ = 7 Hz, —$CH_2CH_3$), 1.34 (t, 3H, $J$ = 7 Hz, —$OCH_2CH_3$), and 0.99 (t, 3H, $J$ = 7 Hz, —$CH_3$) and the minor at δ: 6.04 (br s, 1H, —OH), 5.62 (d, 1H, $J$ = 8 Hz, =CH—), 5.29 (s, 1H, =CHCO—), 4.29 (q, 2H, $J$ = 7 Hz, —$OCH_2$—), 2.09 (q, 2H, $J$ = 7 Hz, —$CH_2CH_3$), 1.36 (t, 3H, $J$ = 7 Hz, —$OCH_2CH_3$), and 0.96 (t, 3H, $J$ = 7 Hz, —$CH_3$). *Anal.* calcd. for $C_{14}H_{18}O_4$: C 67.17, H 7.25; found: C 67.01, H 7.07.

*5-Carbethoxy-9-chloro-3-ethylbicyclo[4.3.0]nona-3,8-dien-7-one (8) and 2-carbethoxy-9-chloro-4-ethylbicyclo[4.3.0]nona-3,8-dien-7-one (9)*

At 0°C, to a solution of diketone **7** (1.03 g, 4.1 mmol) in tetrahydrofuran (15 mL) under a nitrogen atmosphere, were added lithium hydride (43 mg, 5.4 mmol) and hexamethylphosphorous triamide (0.94 mL, 5.2 mmol) with stirring. After 10 min, phenyl dichlorophosphate (1.36 mL, 9.1 mmol) and lithium chloride (380 mg, 9 mmol) were added. The reaction mixture was stirred at room temperature for 16 h. Ice-cold water and dilute hydrochloric acid were added and the resulting mixture was extracted with chloroform. The extracts were washed with water, dried, filtered, and concentrated. Flash chromatography (10) of the residue on silica gel, eluting with 8% ethyl acetate in *n*-hexane, gave a 3:2 mixture (nmr analysis) of chlorides **8** and **9** (707 mg; 64% yield). The mixture was separated by preparative high pressure liquid chromatography on a Waters Associates Prep LC/System 500 using one silica gel cartridge and eluting with 7% ethyl acetate in *n*-hexane. Fractions were collected by shaving the leading and trailing edges of the single peak and recycling the central portion. The combined "leading edge" fractions were concentrated to give pure chloride **8** (178 mg); ir (neat): 1716 (ester and ketone) and 1591 (C=C) $cm^{-1}$; nmr δ: 6.21 (d, 1H, $J$ = 2 Hz, =CHCO—), 5.53 (tdd, 1H, $J$ = 7, $J'$ = 2, $J''$ = 1 Hz, —CH=), 4.15, 4.13 (both q, 1H each, $J$ = 7 Hz each, —$OCH_2$—), 3.61 (dd, 1H, $J$ = 7, $J'$ = 2 Hz, —CHCOO—), 3.51 (dddd, 1H, $J = J'$ = 7, $J''$ = 2,5, $J'''$ = 2Hz, —CHCCl=), 3.23 (dd, 1H, $J$ = 7, $J'$ = 2 Hz, —CHCO—), 2.47 (ddd, 1H, $J$ = 16, $J'$ = 7, $J''$ = 2 Hz, —C*H*H—), 2.29 (dd, 1H, $J$ = 16, $J'$ = 2.5 Hz,—C*H*H—), 1.98, 1.96 (both dq, 1H each, $J$ = 7, $J'$ = 1 Hz each, —$CH_2CH_3$), 1.26 (t, 3H, $J$ = 7 Hz, —$OCH_2CH_3$), and 0.94 (t, 3H, $J$ = 7 Hz, —$CH_3$); ms: $M^+$ 270.0842 and 268.0865 (calcd. for $C_{14}H_{17}ClO_3$: 270.0837 and 268.0865). *Anal.* calcd. for $C_{14}H_{17}ClO_3$: C 62.55, H 6.38, Cl 13.20; found: C 62.50, H 6.58, Cl 12.89. The "trailing edge" fractions were combined and concentratd to give chloride **9** (196 mg); ir (neat): 1716 (ester and ketone) and 1593 (C=C) $cm^{-1}$; nmr δ: 6.23 (d, 1H, $J$ = 2 Hz, =CHCO—), 5.43 (dd, 1H, $J$ = 7, $J'$ = 2 Hz, —CH=), 4.17 (q, 2H, $J$ = 7 Hz, —$OCH_2$—), 3.78 (ddd, 1H, $J$ = 7, $J' = J''$ = 2 Hz, —CHCCl=), 3.53 (dd, 1H, $J$ = 7, $J'$ = 2 Hz, —CHCOO—), 2.96 (ddd, 1H, $J = J'$ = 7, $J''$ = 4 Hz, —CHCO—), 2.32 (m, 2H, —$CH_2$—), 1.99 (q, 2H, $J$ = 7 Hz, —$CH_2CH_3$), 1.28 (t, 3H, $J$ = 7 Hz, —$OCH_2CH_3$), and 0.94 (t, 3H, $J$ = 7 Hz, —$CH_3$); ms: $M^+$ 270.0850 and 268.0875 (calcd. for $C_{14}H_{17}ClO_3$: 270.0837 and 268.0865). The remaining material was recovered as a mixture of **8** and **9**.

*2-Carbethoxy-4-ethylbicyclo[4.3.0]non-3-en-7-one (10) from chloride 9*

To a solution of chloride **9** (48 mg, 0.18 mmol) in benzene (1.2 mL) were added sodium bicarbonate (15 mg, 0.18 mmol) and 5% Pd/C (4.8 mg). After stirring under an atmosphere of hydrogen for 4 h, the reaction mixture was filtered and concentrated. Flash chromatography of the residue on silica gel, eluting with 5% ethyl acetate in *n*-hexane, gave a mixture of epimeric keto esters **10** (35 mg; 83% yield); ir (neat): 1732 (ketone and ester) $cm^{-1}$; nmr δ: 5.44, 5.37 (both br s, ~1:9, total 1H, —CH=), 4.18 (q, 2H, $J$ = 7 Hz, —$OCH_2$—), 1.33, 1.34 (both t, ~9:1, total 3H, $J$ = 7 Hz each, —$OCH_2CH_3$), and 1.03 (t, 3H, $J$ = 7 Hz, —$CH_3$); ms: $M^+$ 236.1410 (calcd. for $C_{14}H_{20}O_3$: 236.1413). *Anal.* calcd. for $C_{14}H_{20}O_3$: C 71.14, H 8.54; found: C 70.85, H 8.54.

*5-Carbethoxy-3-ethyl-9-methoxybicyclo[4.3.0]nona-3,8-dien-7-one (12) and 5-carbomethoxy-3-ethyl-9-methoxybicyclo[4.3.0]nona-3,8-dien-7-one (13)*

Chloride **8** (381 mg, 1.42 mmol) was dissolved in methanol (10 mL) under a nitrogen atmosphere in a flask wrapped in aluminum foil to exclude light. Silver nitrate (482 mg, 2.84 mmol) was introduced and the reaction mixture heated at 70°C for 19 h. Filtration and concentration gave the crude product which was subjected to flash chromatography on silica gel. Elution with 35% ethyl acetate in *n*-hexane gave ethyl ester **12** (276 mg; 74% yield); ir (neat): 1724 (ester), 1698 (ketone), and 1600 (C=C) $cm^{-1}$; nmr δ: 5.53 (tdd, 1H, $J$ = 7, $J'$ = 2, $J''$ = 1 Hz, —CH=), 5.32 (d, 1H, $J$ = 1 Hz, =CHCO—), 4.16, 4.14 (both q, 1H each, $J$ = 7 Hz each, —$OCH_2$—), 3.86 (s, 3H, —$OCH_3$), 3.62 (dd, 1H, $J$ = 7 Hz, $J'$ = 2 Hz, —CHCOO—), 3.33 (dddd, $J = J'$ = 7, $J''$ = 3, $J'''$ = 1 Hz, —C*H*$CH_2$—), 3.15 (dd, 1H, $J$ = 7, $J'$ = 2 Hz, —CHCO—), 2.39 (ddd, 1H, $J$ = 16, $J'$ = 7, $J''$ = 2 Hz, —C*H*H—), 2.24 (dd, 1H, $J$ = 16, $J'$ = 3 Hz, —C*H*H—), 1.98, 1.96 (both dq, 1H each, $J$ = 7, $J'$ = 1 Hz each, —$CH_2CH_3$), 1.27 (t, 3H, $J$ = 7 Hz, —$OCH_2CH_3$), and 0.94 (t, 3H, $J$ = 7 Hz, —$CH_3$); ms: $M^+$ 264.1365 (calcd. for $C_{15}H_{20}O_4$: 264.1362). Further elution with the same solvent system gave methyl ester **13** (24 mg; 8% yield); ir (neat): 1735 (ester) 1695 (ketone), and 1598 (C=C) $cm^{-1}$; nmr δ: 5.56 (br d, 1H, $J$ = 7 Hz, —CH=), 5.30 (s, 1H, =CHCO—), 3.83 (s, 3H, —$OCH_3$), 3.71 (s, 3H, —$COOCH_3$), and 0.93 (t, 3H, $J$ = 7 Hz, —$OCH_2CH_3$); ms: $M^+$ 250.1210 (calcd. for $C_{14}H_{18}O_4$: 250.1205).

*4-Ethyl-2-hydroxymethylbicyclo[4.3.0]nona-3,8-dien-7-one (14)*

At −20°C, to a solution of esters **12** and **13** (11:1; 389 mg, 1.48 mmol) in ether (8 mL) under a nitrogen atmosphere was added lithium aluminum hydride (112 mg, 2.95 mmol). After stirring for 20 min, the reaction mixture was allowed to warm up to −5°C. Water (2 mL) was slowly added. The resulting mixture was acidified with 1 *N* hydrochloric acid, stirred at 0°C for 45 min, and extracted with methylene chloride. The organic solution was dried, filtered, and concentrated. Flash chromatography of the residue on silica gel, eluting with 45% ethyl acetate in *n*-hexane gave alcohol **14** (198 mg; 70% yield); ir (neat): 3425 (OH), 1704 (C=O), and 1585 (C=C) $cm^{-1}$; nmr δ: 7.64 (dd, 1H, $J$ = 6, $J'$ = 3 Hz, —C*H*=CHCO—), 6.17 (dd, 1H, $J$ = 6, $J'$ = 2 Hz, =CHCO—), 5.35 (d, 1H, $J$ = 5 Hz, —CH=), 3.75, 3.69 (both dd, 1H each, $J$ = 18, $J'$ = 6 Hz each, —$CH_2O$—), 2.03 (q, 2H, $J$ = 7 Hz, —$CH_2CH_3$), and 0.98 (t, 3H, $J$ = 7 Hz, —$CH_3$); ms: $M^+$ 192.1143 (calcd. for $C_{12}H_{16}O_2$: 192.1150). *Anal.* calcd. for $C_{12}H_{16}O_2$: C 74.97, H 8.39; found: C 74.78, H 8.57.

*2-Carboxy-4-ethylbicyclo[4.3.0]nona-3,8-dien-7-one (15) and 2-carbethoxy-4-ethylbicyclo[4.3.0]non-3,8-dien-7-one (16)*

At 0°C, to a solution of alcohol **14** (131 mg, 0.68 mmol) in acetone (4 mL) was added dropwise 1 mL of 8 *N* Jones reagent (11) with stirring. After 1 h, the reaction mixture was diluted with water and extracted with ethyl acetate. Drying, filtration, and concentration gave 120 mg of acid **15** (ir ($CHCl_3$ cast): 3060 (acid), 1700 (acid and ketone), and 1590 (C=C) $cm^{-1}$). This compound, without purification, was dissolved in acetone (4 mL) and potassium carbonate (161 mg, 1.17 mmol) was added. The mixture was stirred at room temperature under a nitrogen atmosphere for 1 h and iodoethane

(0.7 mL, 8.75 mmol) was then introduced. After heating at reflux for 16 h, the reaction mixture was poured into ice-cold water and extracted with methylene chloride. The organic solution was dried, filtered, and concentrated. Flash chromatography of the residue on silica gel, eluting with 8% ethyl acetate in *n*-hexane, gave enone ester **16** (103 mg; 65% yield from alcohol **14**); ir ($CH_2Cl_2$ cast): 1730 (ester), 1712 (ketone), 1590 (C=C) $cm^{-1}$; nmr δ: 7.62 (dd, 1H, $J$ = 6, $J'$ = 3 Hz, —C*H*=CHCO—), 6.26 (dd, 1H, $J$ = 6, $J'$ = 2 Hz, =CHCO—), 5.51 (dd, $J$ = 7, $J'$ = 2 Hz, —CH=), 4.22 (q, 2H, $J$ = 7 Hz, —$OCH_2$—), 2.05 (q, 2H, $J$ = 7 Hz, —$CH_2CH_3$), 1.31 (t, 3H, $J$ = 7 Hz, —$OCH_2CH_3$), and 0.99 (q, 3H, $J$ = 7 Hz, —$CH_3$); ms: $M^+$ 234.1256 (calcd. for $C_{14}H_{18}O_3$: 234.1256).

*Keto esters **10** from enone ester **16***

Enone ester **16** (85 mg, 0.36 mmol) was dissolved in ethyl acetate (3 mL) and 5% Pd/C (9 mg) was added. The mixture was stirred under an atmosphere of hydrogen at room temperature for 1 h. Filtration and concentration gave the crude product, which was purified by flash chromatography on silica gel. Elution with 5% ethyl acetate in *n*-hexane gave a mixture of keto esters **10** (78 mg; 92% yield) identical with that obtained previously from chloride **9**.

*2-Carbethoxy-4-ethylbicyclo[4.3.0]non-2-en-7-one (**17**)*

At 0°C, to a solution of keto esters **10** (35 mg, 0.15 mmol) in ethanol (1.5 mL) was added sodium hydride (80% oil dispersion; 10 mg, 0.33 mmol). After stirring at room temperature under a nitrogen atmosphere for 16 h, the reaction mixture was diluted with ice-cold water, acidified with dilute hydrochloric acid, and extracted with methylene chloride. The organic solution was dried, filtered, and concentrated. Flash chromatography of the residue on silica gel, eluting with 5% ethyl acetate in *n*-hexane, gave a mixture of epimeric esters **17** (25 mg; 71% yield); ir ($CHCl_3$ cast): 1742 (ketone), 1711 (ester), and 1642 (C=C) $cm^{-1}$; nmr δ: 6.98, 6.92 (2:5; both br s, total 1H, —CH=), 4.21 (m, 2H, —$OCH_2$—), 1.32, 1.27 (5:2; both t, total 3H, $J$ = 7 Hz each, —$OCH_2CH_3$), 1.01, and 0.99 (5:2; both t, total 3H, $J$ = 7 Hz each, —$CH_3$); ms: $M^+$ 236.1408 (calcd. for $C_{14}H_{20}O_3$: 236.1412). *Anal.* calcd. for $C_{14}H_{20}O_3$: C 71.16, H 8.53; found: C 71.14, H 8.53.

d,l-*Coronafacic acid (**2**)*

A solution of esters **17** (20 mg, 0.085 mmol) in 2.4 *N* aqueous hydrochloric acid (1.5 mL) was heated at reflux under a nitrogen atmosphere for 5.5 h. After cooling to room temperature, the resulting solution was diluted with water (2 mL) and extracted with ethyl acetate. The extracts were dried, filtered, and concentrated. Flash chromatography of the residue on silica gel, eluting with 40% ethyl acetate in *n*-hexane, gave 13 mg (73% yield) of a 4:1 (nmr integration) mixture of coronafacic acid (**2**) and its C-6 epimer. In the nmr spectrum, the vinyl proton of the latter compound appeared as a doublet of doublets ($J$ = 4, $J'$ = 2 Hz) at δ 7.12 in agreement with the reported value (1). Recrystallization of the mixture resulted in partial epimerization of the minor isomer to coronafacic acid (**2**) and gave 11 mg of the latter compound in crystalline form, mp 121–125°C. Concentration of the mother liquid gave 2 mg of a 1:1 mixture of **2** and its C-6 epimer. The ir, nmr, and mass spectra of the synthetic coronafacic acid were found to be identical with those of an authentic sample and displayed the following characteristic features: ir ($CHCl_3$ cast): 3040, 2634, 2532, 1685 (acid), 1741 (ketone), and 1634 (C=C) $cm^{-1}$; nmr δ: 7.06 (br s, 1H, —CH=) and 1.01 (t, 3H, $J$ = 7 Hz, —$CH_3$); ms: $M^+$ 208.1104 (100%; calcd. for $C_{12}H_{16}O_3$: 208.1099).

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