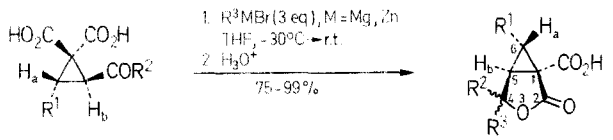


1a-c 2a-h

1	R^1	R^2
a	CH_3	CH_3
b	C_6H_5	CH_3
c	C_6H_5	C_6H_5

2	R^1	R^2	R^3
a	CH_3	CH_3	$H_2C=CH-CH_2$
b	C_6H_5	CH_3	C_2H_5[a]
c	C_6H_5	CH_3	$H_2C=CH-CH_2$
d	C_6H_5	CH_3	$H_2C=C(CH_3)-CH_2$
e	C_6H_5	CH_3	$CH_2=CH-CH(CH_3)$[b]
f	C_6H_5	C_6H_5	C_2H_5[a]
g	C_6H_5	C_6H_5	n-C_3H_7[a]
h	C_6H_5	C_6H_5	$H_2C=CH-CH_2$

[a] M = Mg.
[b] with $CH_3-CH=CH-CH_2MBr$

Scheme A

A Convenient Synthesis of Substituted 3-Oxabicyclo [3.1.0]hexane-2-ones

Margarita MLADENOVA

Institute of Organic Chemistry with Centre of Phytochemistry, Bulgarian Academy of Sciences, 1113 Sofia, Bulgaria

Françoise GAUDEMAR-BARDONE*, Nicole GOASDOUE, Marcel GAUDEMAR

Université Pierre et Marie Curie, Laboratoire de Synthèse Organométallique, Bâtiment F, 4 Place Jussieu, F-75230 Paris Cédex 05, France.

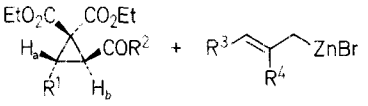
1-Carboxy (or 1-ethoxycarbonyl)-3 oxabicyclo[3.1.0]hexane-2-ones (**2** and **4**) are readily prepared from 2-acyl-3-methyl (or 3-phenyl)-1,1-dicarboxycylcopropanes (**1**) and 2-acyl-3-alkyl (or 3-phenyl)-1,1-diethoxycarbonylcyclopropanes (**3**), respectively, in 70–99% overall yields.

Methods so far reported in the literature for the preparation of γ-lactones fused with a cyclopropane ring system are scarce, often occasional or rather sophisticated, and restricted in scope[1-10].

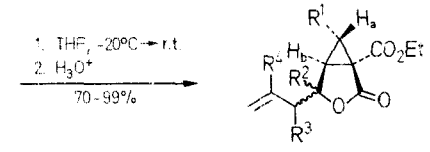
In view of a pharmacological study we have been led to synthesize some examples of the title compounds. By this reason we report in the present paper a simple and convenient synthesis of diversely substituted 1-carboxy (or 1-ethoxycarbonyl)-3-oxabicyclo[3.1.0]hexane-2-ones **2** (or **4**). The key reagents for their preparation are 2-acyl-3-methyl (or 3-phenyl)-1,1-dicarboxycyclopropanes **1** and 2-acyl-3 alkyl (or 3-phenyl)-1,1-diethoxycarbonylcyclopropanes **3**. Some years ago two of us have described an easy synthesis of these cyclopropylic compounds from α,β-unsaturated ketones and the organomagnesium derivative of ethyl dibromomalonate[11]. It is noteworthy that the yields in the synthesis of **3** are obviously improved by using ultra-sound.

Our synthesis of the compounds **2** consists in the condensation of 3 equivalents of a Grignard or an allylzinc reagent with **1** (Scheme **A**).

Compounds **3** react in a similar manner with allylzinc reagents to give 1-ethoxycarbonyl-3-oxabicyclo[3.1.0]hexane-2-ones **4** in good yields (Scheme **B**). Attempts to carry out the reaction with Grignard reagents are unsuccessful.



3a-e



4a-h

3	R^1	R^2
a	CH_3	CH_3
b	n-C_3H_7	CH_3
c	C_6H_5	H
d	C_6H_5	CH_3
e	C_6H_5	C_6H_5

4	R^1	R^2	R^3	R^4
a	CH_3	CH_3	H	CH_3
b	n-C_3H_7	CH_3	H	H
c	C_6H_5	H	H	H
d	C_6H_5	CH_3	H	H
e	C_6H_5	CH_3	H	CH_3
f	C_6H_5	C_6H_5	H	H
g	C_6H_5	C_6H_5	H	CH_3
h	C_6H_5	C_6H_5	CH_3	H

Scheme B

Table 1. 1-Carboxy-3-oxabicyclo[3.1.0]hexane-2-ones (**2**) Prepared

Product **2**	Yield [%]	*cis*/*trans* (**A**/**B**) ratio	m.p. [°C] (solvent)	Molecular Formula[a]	Isomer	IR (KBr)[b] ν [cm^{-1}]	^{1}H-NMR ($CDCl_3$/TMS)[c] δ [ppm]
a	90	>95 : 5	86–87 (hexane)	$C_{11}H_{14}O_4$ (210.2)	*cis*	1755, 1730	1.39 (d, 3H, CH_3); 1.47 (s, 3H, CH_3); 2.03 (m, H_a, CH); 2.40 and 2.52 (dd, 2H, J = 14.2 Hz, 7.6 Hz, CH_2); 2.55 (d, H_b, J = 5.7 Hz, CH); 5.20 and 5.25 (m, 2H, J = 11.0 Hz, 1.5 Hz, H_2C=); 5.8 (m, 1H, J = 11.0 Hz, 7.6 Hz, =CH)
b	88	63 : 37	oil	$C_{15}H_{16}O_4$ (260.3)	*cis*	1760, 1720	1.07 (t, 3H, CH_3); 1.56 (s, 3H, CH_3); 1.80–1.92 (m, 2H, CH_2); 3.12 (d, H_a, J = 6.0 Hz, CH); 3.25 (d, H_b, J = 6.0 Hz, CH); 7.20–7.40 (m, 5H, C_6H_5)[e]
					trans		1.03 (t, 3H, J = 7.5 Hz, CH_3); 1.50 (s, 3H, CH_3); 1.70–1.85 (m, 2H, CH_2); 3.09 (d, H_a, J = 5.9 Hz, CH); 3.21 (d, H_b, J = 5.9 Hz, CH)[e]
c	76	83 : 17	125–126[d] ($CHCl_3$/hexane)	$C_{16}H_{16}O_4$ (272.3)	*cis*	1760, 1715, 1690	1.58 (s, 3H, CH_3); 2.50 (dd, 1H, J = 13.8 Hz, 7.8 Hz, HCH); 2.61 (dd, 1H, J = 13.8 Hz, 6.8 Hz, HCH); 3.19 (d, H_a, J = 5.8 Hz, CH); 3.24 (d, H_b, J = 5.8 Hz, CH); 5.25–5.31 (m, 2H, H_2C=); 5.75–5.86 (m, 1H, =CH); 7.25–7.40 (m, 5H, C_6H_5)
					trans		1.55 (s, 3H, CH_3); 2.60 (s, 2H, CH_2)[e]
d	99	17 : 83		$C_{17}H_{18}O_4$ (286.3)	*cis*		1.55 (s, 3H, CH_3); 1.85 (s, 3H, CH_3); 2.50 (d, 1H, J = 14.0 Hz, HCH); 2.64 (d, 1H, J = 14.0 Hz, HCH); 4.90 (br. s, 1H, HCH=); 5.06 (br. s, 1H, HCH=)[e]
			131–132[d] ($CHCl_3$/hexane)		*trans*	1780, 1695	1.57 (s, 3H, CH_3); 1.82 (s, 3H, CH_3); 2.52 (s, 2H, CH_2); 3.20 (d, H_a, J = 6.0 Hz, CH); 3.28 (d, H_b, J = 6.0 Hz, CH); 4.88 (br. s, 1H, HCH=); 5.00 (br. s, 1H, HCH=); 7.25–7.36 (m, 5H, C_6H_5)
e	90	60 : 40 (**A**/**B**)	127–133 (60/40)	$C_{17}H_{18}O_4$ (286.3)	**A**	1780, 1690	1.20 (d, 3H, CH_3); 1.46 (s, 3H, CH_3); 2.60 (m, 1H, CH); 3.25 (d, H_a, J = 6.0 Hz, CH); 3.27 (d, H_b, J = 6.0 Hz, CH); 5.17 (d, 1H, J = 18.5 Hz, HCH=); 5.22 (d, 1H, J = 11.9 Hz, HCH=); 5.92 (m, 1H, =CH); 7.24–7.40 (m, 5H, C_6H_5)[e]
					B		1.19 (d, 3H, CH_3); 1.48 (s, 3H, CH_3); 5.85 (m, 1H, =CH)[e]
f	75	<5 : 95	135–136 (CCl_4)	$C_{20}H_{18}O_4$ (322.3)	*trans*	1765, 1705	0.85 (t, 3H, CH_3); 2.15 (q, 2H, J = 7.4 Hz, CH_2); 3.33 (d, H_a, J = 6.0 Hz, CH); 3.65 (d, H_b, J = 6.0 Hz, CH); 7.23–7.53 (m, 10H, $2C_6H_5$)
g	78	<5 : 95	178–179 (acetone/hexane)	$C_{21}H_{20}O_4$ (336.4)	*trans*	1760, 1720	0.85 (t, 3H, CH_3); 1.03–1.44 (m, 2H, CH_2); 2.08 (m, 2H, CH_2); 3.34 (d, H_a, J = 6.0 Hz, CH); 3.64 (d, H_b, J = 6.0 Hz, CH); 7.21–7.52 (m, 10H, $2C_6H_5$)
h	75	<5 : 95	124–125 (ether/hexane)	$C_{21}H_{18}O_4$ (334.3)	*trans*	1765, 1725, 1695	2.88 (dd, 2H, J = 7.1 Hz, 1 Hz, CH_2); 3.38 (d, H_a, J = 6.0 Hz, CH); 3.65 (d, H_b, J = 6.0 Hz, CH); 5.07 (dd, 1H, J = 18.9 Hz, 2.9 Hz, HCH=); 5.10 (dd, 1H, J = 10.4 Hz, 1.6 Hz, HCH=); 5.49–5.79 (m, 1H, =CH); 7.15–7.45 (m, 10H, $2C_6H_5$)

[a] Satisfactory microanalyses obtained: C ±0.32, H ±0.28.
[b] Perkin-Elmer R257 spectrophotometer.
[c] Recorded at 250 MHz on a Bruker WM spectrometer.
[d] Separated by crystallization from the mixture of the two isomers.
[e] Values from the spectrum of the mixture of the two isomers.

Table 2. 1-Ethoxycarbonyl-3-oxabicyclo[3.1.0]hexane-2-ones (**4**) Prepared

Product **4**	Yield [%]	*cis*/*trans* ratio	m.p. [°C] or b.p. [°C]/torr (solvent)	Molecular Formula[a]	Isomer	IR (KBr)[b] ν [cm^{-1}]	^{1}H-NMR ($CDCl_3$/TMS)[c] δ [ppm]
a	75	–:100	100–102/0.05	$C_{14}H_{20}O_4$ (252.3)	*trans*	1785, 1730	1.32 (d, 3H, CH_3); 1.33 (t, 3H, CH_3); 1.43 (s, 3H, CH_3); 1.81 (s, 3H, CH_3); 1.80–1.88 (m, H_a, CH); 2.36 (s, 2H, CH_2); 2.31–2.46 (m, H_b, CH); 4.28 (q, 4H, CH_2); 4.83, 4.95 (2s, 2H, $H_2C=$)
b	98	83:17	122–123/0.05	$C_{15}H_{22}O_4$ (266.3)	*cis*	1785, 1730	0.94 (t, 3H, CH_3); 1.33 (t, 3H, CH_3); 1.43 (s, 3H, CH_3); 1.38–1.70 (m, 4H, $2CH_2$); 1.76 (m, H_a, J = 5.5 Hz, CH); 2.36 (d, H_b, J = 5.5 Hz, CH); 2.34 (dd, 1H, J = 14.0 Hz, 5.6 Hz, HCH); 2.44 (dd, 1H, J = 14.0 Hz, 7.0 Hz, HCH); 5.18 (d, 1H, J = 14.8 Hz, HCH=); 5.19 (d, 1H, J = 11.4 Hz, HCH=); 5.68–5.85 (m, 1H, =CH)[c]
					trans		1.32 (t, 3H, J = 7.1 Hz, CH_3); 1.37 (s, 3H, CH_3)[c]
c	70	55:45	161/0.07	$C_{17}H_{18}O_4$ (286.3)	*cis*	1785, 1730	0.92 (t, 3H, CH_3); 2.42–2.64 (m, 2H, CH_2); 3.00 (d, H_a, J = 5.7 Hz, CH); 3.26, 3.28 (dd, H_b, J = 5.58 Hz, 5.57 Hz, CH); 3.88–4.02 (m, 2H, CH_2); 4.74–4.81 (m, H, CH); 5.21–5.31 (m, 2H, $H_2C=$); 5.77–5.94 (m, H, =CH); 7.21–7.39 (m, 5H, C_6H_5)
					trans		0.91 (t, 3H, CH_3); 2.59 (m, 2H, CH_2); 2.89 (d, H_a, J = 5.5 Hz, CH); 3.15 (d, H_b, J = 5.5 Hz, CH); 4.55 (t, H, J = 6.2 Hz, CH)[c]
d	98	89:11	83–84[d] ($CHCl_3$/hexane)	$C_{18}H_{20}O_4$ (300.3)	*cis*	1780, 1720	0.91 (t, 3H, CH_3); 1.56 (s, 3H, CH_3); 2.44 (dd, 1H, J = 13.7 Hz, 7.7 Hz, HCH); 2.56 (dd, 1H, J = 13.7 Hz, 6.9 Hz, HCH); 3.02 (d, H_a, J = 5.7 Hz, CH); 3.06 (d, H_b, J = 5.7 Hz, CH); 3.96 (dq, 2H, J = 7.2 Hz, 2.6 Hz, CH_2); 5.20 (d, 1H, J = 17.5 Hz, HCH=); 5.22 (d, 1H, J = 8.8 Hz, HCH=); 5.75–5.95 (m, 1H, =CH); 7.21–7.39 (m, 5H, C_6H_5)
					trans		0.93 (t, 3H, CH_3); 1.48 (s, 3H, CH_3); 2.59 (d, 2H, J = 6.9 Hz, CH_2); 3.00 (d, H_a, J = 5.6 Hz, CH); 3.10 (d, H_b, J = 5.6 Hz, CH); 3.96 (dq, 2H, J = 7.2 Hz, 2.6 Hz, CH_2); 5.23–5.30 (m, 2H, $H_2C=$); 5.75–5.95 (m, 1H, =CH); 7.22–7.36 (m, 5H, C_6H_5)[c]
e	99	8:92		$C_{19}H_{22}O_4$ (314.4)	*cis*		0.93 (t, 3H, CH_3); 1.48 (s, 3H, CH_3); 1.87 (s, 3H, CH_3); 2.49 (d, 1H, J = 13.5, HCH); 2.60 (d, 1H, J = 13.5 Hz, HCH); 4.90 (br. s, 1H, HCH=); 5.02 (br. s, 1H, HCH=)[c]
			39–41[d] ($CHCl_3$/hexane)		*trans*	1785, 1725	0.91 (t, 3H, CH_3); 1.55 (s, 3H, CH_3); 1.83 (s, 3H, CH_3); 2.46 (s, 2H, CH_2); 3.03 (d, H_a, J = 5.7 Hz, CH); 3.11 (d, H_b, J = 5.7 Hz, CH); 3.95 (m, 2H, CH_2); 4.86 (br. s, 1H, HCH=); 4.97 (br. s, 1H, HCH=); 7.23–7.33 (m, 5H, C_6H_5)
f	98	100:–	108–109 ($CHCl_3$/hexane)	$C_{23}H_{22}O_4$ (362.4)	*cis*	1780, 1730	0.82 (t, 3H, CH_3); 2.85 (d, 2H, CH_2); 3.26 (d, H_a, J = 5.8 Hz, CH); 3.52 (d, H_b, J = 5.8 Hz, CH); 3.89 (q, 2H, CH_2); 5.06 (d, 1H, J = 16.9 Hz, HCH=); 5.08 (d, 1H, J = 9.2 Hz, HCH=); 5.52–5.69 (m, 1H, =CH); 7.28–7.55 (m, 10H, $2C_6H_5$)

Table 2. (Continued)

Product 4	Yield [%]	*cis*/*trans* ratio	m.p. [°C] or b.p. [°C]/torr (solvent)	Molecular Formula[a]	Isomer	IR (KBr)[b] ν[cm^{-1}]	^{1}H-NMR ($CDCl_3$/TMS)[c] δ[ppm]
g	98	–:100	142–143 ($CHCl_3$/hexane)	$C_{25}H_{24}O_4$ (376.4)	*trans*	1780, 1725	0.85 (t, 3H, CH_3); 1.52 (s, 3H, CH_3); 2.77 (d, 1H, J = 14.1 Hz, HCH); 2.87 (d, 1H, J = 14.1 Hz, HCH); 3.26 (d, H_a, J = 5.7 Hz, CH); 3.57 (d, H_b, J = 5.7 Hz, CH); 3.87 (q, 2H, CH_2); 4.62 (br. s, 1H, HCH=); 4.78 (br. s, 1H, HCH=); 7.26–7.53 (m, 10H, $2C_6H_5$)
h	85	65:35 (*trans* **A**/ *trans* **B**)	145–146[d] ($CHCl_3$/hexane)	$C_{24}H_{24}O_4$ (376.4)	*trans* **A**	1780, 1725	0.83 (t, 3H, CH_3); 1.14 (d, 3H, CH_3); 2.82 (m, H, CH); 3.32 (d, H_a, J = 5.8 Hz, CH); 3.61 (d, H_b, J = 5.8 Hz, CH); 3.85 (q, 2H, CH_2); 4.98–5.04 (m, 2H, H_2C=); 5.53–5.85 (m, H, =CH); 7.25–7.55 (m, 10H, $2C_6H_5$)
					trans **B**		0.81 (t, 3H, CH_3); 0.90 (d, 3H, CH_3); 3.32 (d, H_a, J = 5.6 Hz, CH); 3.58 (d, H_b, J = 5.6 Hz, CH); 3.82 (q, 2H, CH_2); 5.04–5.27 (m, 2H, H_2C=)[e]

[a] Satisfactory microanalyses obtained: C ±0.30, H ±0.25.
[b,c,d,e] See Table 1.

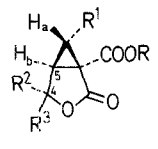
Under our experimental conditions the lactonization probably takes place *in situ* (already observed in another cases[12]) since the NMR spectrum of the crude product shows no trace of the corresponding acyclic compound. Thus, we have prepared two series of bicyclic lactones, **2** (Table 1) and **4** (Table 2), which, as far as we know, have not previously been described. Their structures are established by microanalyses and spectroscopic data.

The following remarks can be made concerning the stereochemistry of compounds **2** and **4**:

– The *cis*-configuration at the centres C_1,C_5 results from the lactone ring closure leading to the *cis*-fused 3-oxabicyclo[3.1.0]hexane-2-one system.

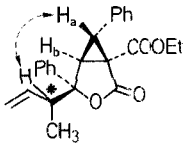
– The *trans*-position of H_a (C_6) and H_b (C_5) derives from the *trans*-configuration of the starting materials **1** and **3**; this configuration, which has been established photochemically[11], is confirmed now by a N.O.E. experiment. Hence, it follows that the substituent R_1 is always in the *exo*-position.

– The relative configuration at C_4 is still to be determined. We name a *cis*-isomer that isomer in which the smallest substituents (according to the sequence rules) at positions 4 and 5 are on the same side of the lactonic ring (H_b and CH_3 when $R^2 = CH_3$ or H_b and R^3 when $R^2 = C_6H_5$) as shown in Fig. 1. The *cis*/*trans* ratio of the isomers is estimated, from the NMR spectra, by means of the integration curve refered to each signal corresponding either to $CH_3 = R^2$ (**2b, c, d, e** and **4b, d, e**) or to H_a or H_b (**4c**), or to H_2C= (**4h**). Assignment of the configuration at C_4 is well established by means of N.O.E.



R = H or C_2H_5; R^2 = CH_3 : *cis* or Ph : *trans*

Fig. 1



4h - *trans*

N.O.E. 2°%(for both diastereomers)

Fig. 2

experiments for *cis*-**2c** (22% enhancement of H_b – integration signal when R^2 is irradiated) and **2f** and **2g** (20%, resp. 22% enhancement of H_a – integration signal when CH_2 of R_3 is irradiated). For compounds *cis*-**2c** and *trans*-**2h** (Fig. 1) a strong magnetic nonequivalence of the diastereotopic methylene protons adjacent to C_4 is observed. A similar magnetic nonequivalence is noted for compounds *cis*-**4c, d** whose configuration at C_4 has been also unambiguously established with N.O.E. measurements. When both isomers are synthesized (for example **4c, d, e**) the *trans*-isomer does not exhibit such magnetic nonequivalence of the methylene protons adjacent to the centre C_4. This different magnetic behavior of the methylene protons depending on the position of allylic substituent with regard to the cyclopropane ring, observed already in the case of bicyclo[4.1.0]heptane-4-one[13], has been chosen as criterium of assignment of the configuration for compounds **2a, d, h** and **4a, b, e, g**.

– In the case of **4h**, as the crotylzinc bromide $CH_3-CH=CH-CH_2ZnBr$ reacts with **1** with complete allylic rearrangement[14], a new asymmetric centre is created in the allylic chain. A N.O.E. experiment shows that the configuration at C_4, C_5 is always *trans*, and the observation of two diastereoisomers is due to this new asymmetric centre (Fig. 2).

– For compounds **2b** and **2e** the overlap of the H_a and H_b-signals in both isomers precludes the determination of the configuration of C_4 by N.O.E. experiments. As the two-dimensional (2D-) NMR spectroscopy provides powerful technique to evidence specific long range coupling, we have assigned the configuration of **2b** by means of this technique. The configuration of **2e** is not assigned with certainty and for this reason we have designed the two isomers by **A** and **B**.

– As seen in Tables 1 and 2, in most cases the creation of the chiral centre at C_4 proceeds with good stereoselectivity. The variation of reaction time or of solvent polarity does not improve it. All melting and boiling points are uncorrected. The melting points are measured using a Kofler hot-stage apparatus. Tetrahydrofuran is freshly distilled from lithium aluminum hydride. All reactions are performed under a nitrogen atmosphere in a 100 ml flask equipped with a thermometer, mechanical stirrer and a pressure equalizing addition funnel. All allylzinc reagents are prepared according to Refs. 14 and 15.

6-*exo*-Methyl(or -phenyl)-1-carboxy-4-methyl(or -phenyl)-4-alkenyl-(or -alkyl)-3-oxabicyclo[3.1.0]hexane-2-ones (2); General Procedure: To a cooled solution of Grignard or allylzinc reagents (30 mmol) in tetrahydrofuran (20 ml) is added dropwise at –30 °C a solution of **1** (7.5 mmol) in tetrahydrofuran (10 ml). The reaction is exothermic. The cooling bath is removed, and the reaction mixture is stirred at

room temperature during 60 min (excepting for **2b** – 15 min and **2g** – 24 hours), then poured into ice-cooled water (40 ml) containing hydrochloric acid (3 ml) and extracted with ether (3 × 20 ml). The combined extracts are washed with saturated aqueous ammonium sulfate solution (2 × 10 ml), then with water (2 × 10 ml) and dried with magnesium sulfate. The solution is concentrated and the residue is purified by recrystallization.

6-*exo*-Alkyl(or -phenyl)-1-ethoxycarbonyl-4-alkenyl-4-methyl(or -phenyl)-3-oxabicyclo[3.1.0]hexane-2-ones (4); General Procedure: A solution of 16 mmol (or 12 mmol for **4e**) of **3** in tetrahydrofuran (5 ml) is added to a stirred solution of allylzinc reagent (20 mmol) in tetrahydrofuran (14 ml) at −20 °C. Stirring is continued for 30 min at room temperature. Work-up as for compounds **2**. Purification by distillation under reduced pressure or recrystallization.

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