

# Cyclodimerization of the Stevens Rearrangement Products from 4-Alkoxy-2-butynyl(alkoxycarbonylmethyl)dimethylammonium Salts into Cyclohexene Derivatives

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**Abstract**—4-Alkoxy-2-butynyl(alkoxycarbonyl)dimethylammonium salts undergo 3,2-Stevens rearrangement by the action of the corresponding sodium alkoxides. Hydrolysis of the rearrangement products with dilute hydrochloric acid gives 3-alkoxy-2-oxo-3-pentenoic acid esters which are converted into 3-methylene-2-oxo-4-pentenoates via 1,4-elimination of alcohol. [2+4]-Cyclodimerization of 3-methylene-2-oxo-4-pentenoates leads to 1,4-bis(alkoxalyl)-4-vinylcyclohexenes.

It is known that Stevens rearrangement of dimethylammonium salts containing an alkoxycarbonyl group together with a 3-substituted 2-propynyl group leads to formation of 3-substituted alkyl 2-dimethylamino-2,4-pentadienoates. Treatment of the latter with dilute hydrochloric acid gives 3-substituted 2-oxo-3-pentenoic acid esters [1] which are converted into 4-substituted 3-hydroxy-5-methyl-5*H*-furanones by the action of concentrated hydrochloric acid. It should be noted that the 3,2-Stevens rearrangement of ammonium salts containing ethyl and higher alkyl groups on the nitrogen is accompanied by intramolecular 1,5-hydride shift leading to the corresponding hydrogenated amino and oxo esters [2–4].

The present communication reports on the results of our study of the Stevens rearrangement occurring by the action of sodium alkoxides on ammonium salts **Ia–Id** which possess 4-alkoxy-2-butynyl group as migrating fragment. Taking into account the hydride mobility of hydrogen in ethers [5, 6], it was important to elucidate whether products of the 2,3-Stevens rearrangement in salts **Ia–Id** undergo hydride ion transfer from the methylene group of the ether fragment (C<sup>4</sup>). In order to exclude hydride shift with participation of methylene groups at the nitrogen atom [4, 5], as subjects for study we selected dimethylammonium salts **Ia–Id** (Scheme 1; Tables 1, 2).

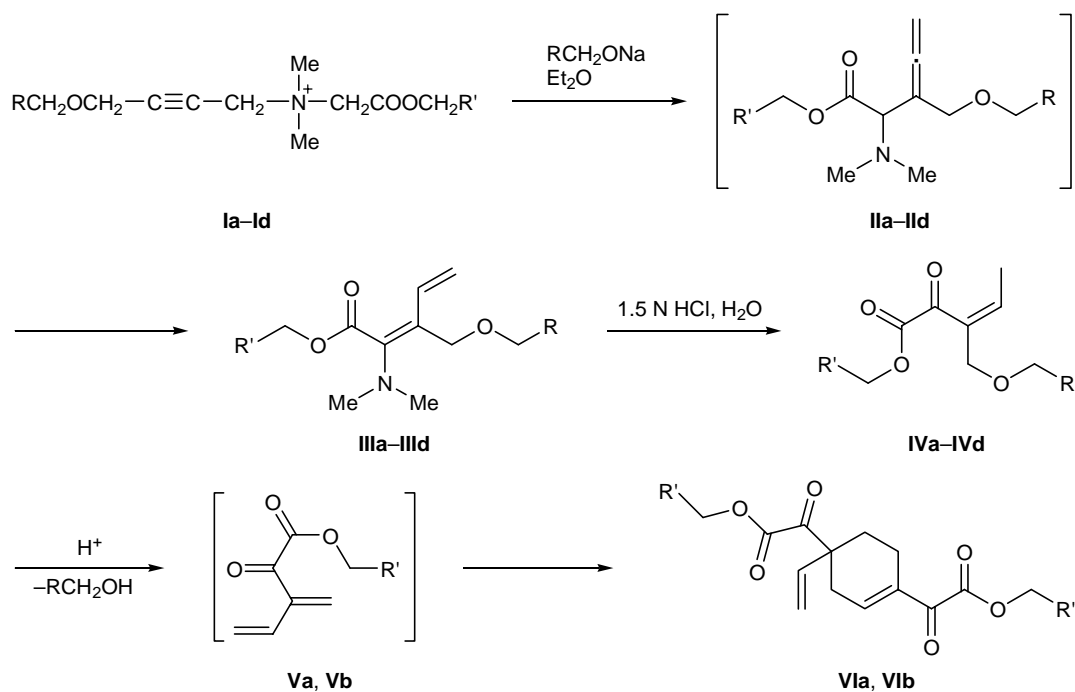
We have found that the Stevens rearrangement of ammonium salts **Ia–Id** does not stop at the stage of

formation of amino esters **IIa–IIId** having an allene structure. The latter undergo prototropic allene–diene isomerization to give 3-alkoxymethyl-2-dimethylamino-2,4-pentadienoates **IIIa–IIId**. Treatment of esters **IIIa–IIId** with dilute hydrochloric acid at 15–17°C affords keto esters **IVa–IVd** which are converted into 3-methylene-2-oxo-4-pentenoates **Va** and **Vb** via 1,4-elimination of the corresponding alcohol. Dimerization of **Va** and **Vb** according to the Diels–Alder pattern [7, 8] yields cyclohexene derivatives **VIa** and **VIb** (Scheme 1, Table 3). It should be emphasized that no hydride ion transfer occurs in rearrangement products **IIa–IIId**.

Both ionic and radical intermediate states were proposed for the Diels–Alder reaction. In keeping with the Woodward–Hoffmann rules, concerted processes are characterized by conservation of orbital symmetry, and such reactions are generally stereoselective [9]. Taking into account published data, we can state with certainty that 1,4-elimination of alcohol molecule from compounds **IVa–IVd** gives *s-cis*-dienes **Va** and **Vb**, for *s-trans*-dienes are known to be inactive in Diels–Alder reactions [7].

The structure of the products was confirmed by the <sup>1</sup>H NMR data, and their purity was checked by GLC. Compounds **VIa** and **VIb** were analyzed by <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy using COSY, DEPT, HMQC, and NOESY techniques, which allowed us to unambiguously assign the corresponding signals. In

Scheme 1.



**I-IV**, R = Me, R' = H (a); R = R' = Me (b); R = Et, R' = H (c); R = Et, R' = Me (d); **V, VI**, R' = H (a); R' = Me (b); **Ia-Ic**, X = Br; **Id**, X = Cl.

the  $^{13}\text{C}$  NMR spectrum of **VIa** we observed four signals from carbonyl carbon atoms, two of which are typical of ketone group, and the other two, of ester.

## EXPERIMENTAL

The IR spectra were recorded on UR-20 and Specord IR-75 instruments. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were obtained on a Varian Mercury-300 spectrometer at 300.075 and 75.46 MHz, respectively, from solutions in  $\text{DMSO-}d_6\text{-CCl}_4$  (1:3) and  $\text{CDCl}_3$  (303 K). The chemical shifts were measured relative to TMS as internal reference. GLC analysis was performed on an LKhM-80 chromatograph equipped with a thermal conductivity detector; 2000 $\times$ 3-mm column packed with 10% of Apieson L on Inerton-AW (0.20–

0.25 mm); oven temperature 50–220 $^\circ\text{C}$  (16 deg/min); carrier gas helium (60 ml/min).

**Ammonium salts Ia-Id** were synthesized by reaction in dry ether of equimolar amounts of the corresponding alkyl bromo(chloro)acetate and (4-alkoxy-2-butynyl)dimethylamine which was prepared according to Mannich [8, 10]. The salts were washed with anhydrous ether and dried in a desiccator over  $\text{CaCl}_2$ . Their yields, melting points, elemental analyses, and  $^1\text{H}$  NMR spectra are given in Tables 1 and 2. The IR spectra of salts **Ia-Id** contained the following absorption bands,  $\nu$ ,  $\text{cm}^{-1}$ : 1050, 1100, 1200, 1240, 1740 ( $\text{COOR}$ ); stretching vibrations of the  $\text{C}=\text{C}$  bonds were almost not observed [11].

**Rearrangement of 4-ethoxy-2-butynyl(methoxy-carbonylmethyl)dimethylammonium bromide (Ia).**

**Table 1.** Yields, melting points, and elemental analyses of ammonium salts **Ia-Id**

Comp. no.	Yield, %	mp, $^\circ\text{C}$	Found, %		Formula	Calculated, %	
			N	Br (Cl)		N	Br (Cl)
<b>Ia</b>	96	87	4.96	28.36	$\text{C}_{10}\text{H}_{20}\text{BrNO}_3$	5.10	28.50
<b>Ib</b>	93	85	4.73	27.02	$\text{C}_{11}\text{H}_{22}\text{BrNO}_3$	4.55	27.35
<b>Ic</b>	94	82	4.52	25.81	$\text{C}_{11}\text{H}_{22}\text{BrNO}_3$	4.75	25.52
<b>Id</b>	96.5	80	5.01	12.70	$\text{C}_{12}\text{H}_{24}\text{ClNO}_3$	5.25	12.54

**Table 2.**  $^1\text{H}$  NMR spectra of ammonium salts **Ia–Id** in  $\text{DMSO-}d_6\text{-CCl}_4$  (1:3),  $\delta$ , ppm ( $J$ , Hz)

Comp. no.	$\text{NMe}_2$ , s	$\text{NCH}_2\text{C}\equiv\text{C}$ , t ( $J = 9$ )	$\text{OCH}_2\text{C}\equiv\text{C}$ , t ( $J = 1.9$ )	$\text{NCH}_2\text{COO}$ , s	$\text{OCH}_2\text{R}$	R	R'
<b>Ia</b>	3.49	4.89	4.25	4.87	3.53 q, $J = 7.0$	1.19 t, $J = 7.0$	3.85 s
<b>Ib</b>	3.49	4.87	4.25	4.82	3.53 q, $J = 7.0$	1.19 t, $J = 7.0$	1.35 t ( $\text{CH}_3$ , $J = 7.2$ ), 4.30 q ( $\text{CH}_2$ , $J = 7.2$ )
<b>Ic</b>	3.50	4.91	4.25	4.90	3.43 t, $J = 6.5$	0.92 t ( $\text{CH}_3$ , $J = 7.4$ ), 1.57 q.t ( $\text{CH}_2$ , $J = 7.4, 6.5$ )	3.84 s
<b>Id</b>	3.49	4.94	4.24	4.92	3.43 t, $J = 6.5$	0.93 t ( $\text{CH}_3$ , $J = 7.4$ ), 1.58 q.t ( $\text{CH}_2$ , $J = 7.4, 6.5$ )	1.35 t ( $\text{CH}_3$ , $J = 7.2$ ), 4.29 q ( $\text{CH}_2$ , $J = 7.2$ )

**Table 3.** Yields, constants, and elemental analyses of compounds **IVa–IVd**, **VIa**, and **VIb** obtained by rearrangement of salts **Ia–Id** by the action of a suspension of sodium methoxide (ethoxide) in diethyl ether

Comp. no.	Yield, <sup>a</sup> %	bp, °C ( $p$ , mm)	$n_D^{20}$	Found, %		Formula	Calculated, %	
				C	H		C	H
<b>IVa</b>	21.4	88–90 (1)	1.4542	57.63	7.11	$\text{C}_9\text{H}_{14}\text{O}_4$	58.06	7.53
<b>IVb</b>	19.8	110–112 (1)	1.4886	59.40	8.45	$\text{C}_{10}\text{H}_{16}\text{O}_4$	60.00	8.00
<b>IVc</b>	15	112–114 (2)	1.4576	59.55	7.67	$\text{C}_{10}\text{H}_{16}\text{O}_4$	60.00	8.00
<b>IVd</b>	16.4	114–117 (1)	1.4640	62.05	8.03	$\text{C}_{11}\text{H}_{18}\text{O}_4$	61.68	8.41
<b>VIa</b>	33.6	172–173 (1)	1.5112	60.48	6.29	$\text{C}_{19}\text{H}_{16}\text{O}_6$	60.00	5.71
<b>VIb</b>	31.4	191–193 (2)	1.5075	61.78	6.84	$\text{C}_{16}\text{H}_{20}\text{O}_6$	62.34	6.49
<b>VIa</b>	25	190–193 (2)	1.5070	60.53	6.33	$\text{C}_{19}\text{H}_{16}\text{O}_6$	60.00	5.71
<b>VIb</b>	30	194–197 (1)	1.4960	61.88	6.07	$\text{C}_{16}\text{H}_{20}\text{O}_6$	62.34	6.49

<sup>a</sup> The yields of cyclohexene derivatives **VIa** and **VIb** were calculated on oxo esters **IVa–IVd**.

Sodium methoxide, 0.08 mol, was added to a mixture of 12 g (0.04 mol) of ammonium salt **Ia** and 15 ml of anhydrous diethyl ether. The mixture was intermittently stirred and ground. When the exothermic reaction was over, the mixture was heated for 20 min under reflux, and water and ether were added. The organic layer was separated, and the aqueous layer was extracted with two portions of diethyl ether. The combined extracts were dried over magnesium sulfate, the solvent was distilled off, and the residue was distilled under reduced pressure to isolate 3.9 g (46%) of methyl 3-ethoxymethyl-2-dimethylamino-2,4-pentadienoate (**IIIa**), bp 81–83°C (2 mm),  $n_D^{20} = 1.4892$ . IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 920, 990, 1590, 1625, 3090 ( $\text{CH}=\text{CH}_2$ ), 1080, 1230, 1715 (COO).  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm ( $J$ , Hz): 1.04 t (3H,  $\text{CH}_3\text{CH}_2$ ), 2.32 s (6H,  $\text{NCH}_3$ ), 3.42 q (2H,  $\text{OCH}_2$ ,  $J = 7.3$ ), 3.56 s (3H,  $\text{OCH}_3$ ), 4.19 s (2H,  $=\text{CCH}_2\text{O}$ ), 4.9–5.4 m (2H,  $=\text{CH}_2$ ), 6.2–7.1 m (1H,  $=\text{CH}$ ). Found, %: C 61.43; H 8.51.  $\text{C}_{11}\text{H}_{19}\text{NO}_3$ . Calculated, %: C 61.97; H 8.92.

**Rearrangement of 4-ethoxy-2-butynyl(ethoxy-carbonylmethyl)dimethylammonium bromide (Ib).** The procedure was the same as above. From 12 g (0.039 mol) of ammonium salt **Ib** and 0.078 mol of sodium ethoxide we obtained 2.83 g (32%) of ethyl 3-ethoxymethyl-2-dimethylamino-2,4-pentadienoate (**IIIb**), bp 91–92°C (2 mm),  $n_D^{20} = 1.4896$ . IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 920, 990, 1585, 1630, 3100 ( $\text{CH}=\text{CH}_2$ ), 1070, 1240, 1725 (COO).  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm ( $J$ , Hz): 1.09 t (3H,  $\text{CH}_3\text{CH}_2\text{O}$ ,  $J = 7.2$ ), 1.15 t (3H,  $\text{CH}_3\text{CH}_2\text{OCO}$ ,  $J = 7.2$ ), 2.61 s (6H,  $\text{NCH}_3$ ), 3.53 q (2H,  $\text{CH}_3\text{CH}_2\text{OCO}$ ), 4.14 q (2H,  $\text{CH}_3\text{CH}_2\text{O}$ ,  $J = 7$ ), 4.25 s (2H,  $=\text{CCH}_2\text{O}$ ), 5.02–5.42 m (2H,  $=\text{CH}_2$ ), 6.64 m (1H,  $=\text{CH}$ ). Found, %: C 62.85; H 9.57.  $\text{C}_{11}\text{H}_{19}\text{NO}_3$ . Calculated, %: C 63.44; H 9.25.

**Rearrangement of ammonium salts Ia–Id by the action of a suspension of sodium methoxide (ethoxide) in diethyl ether.** Sodium methoxide or ethoxide, 0.04 mol, was added to 0.02 mol of ammonium salt **Ia–Id** in 15–20 ml of anhydrous diethyl

ether. The procedure was the same as described above for salts **Ia** and **Ib** with the difference that the combined extracts were treated with 1.5 N hydrochloric acid. The organic layer was separated, and the aqueous layer was extracted with two portions of diethyl ether. The extracts were combined, dried over magnesium sulfate, and distilled. The yields, physical constants, and elemental analyses of products **IVa–IVd**, **VIa**, and **VIb** are given in Table 3.

**Methyl 3-ethoxymethyl-2-oxo-3-pentenoate (IVa).** IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 820, 890, 1625, 3020, 3090 ( $\text{CH}=\text{C}$ ), 1080, 1185, 1240, 1735 ( $\text{COO}$ ), 1690 ( $\text{C}=\text{O}$ ).  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm ( $J$ , Hz): 1.14 t (3H,  $\text{OCH}_2\text{CH}_3$ ,  $J = 7.0$ ), 2.02 d (3H,  $=\text{CHCH}_3$ ,  $J = 7.1$ ), 3.45 q (2H,  $\text{OCH}_2\text{CH}_3$ ,  $J = 7.0$ ), 3.83 s (3H,  $\text{OCH}_3$ ), 4.24 s (2H,  $=\text{CCH}_2\text{O}$ ), 6.98 q (1H,  $=\text{CH}$ ,  $J = 7.1$ ).

**Ethyl 3-ethoxymethyl-2-oxo-3-pentenoate (IVb).** IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 820, 890, 1625, 3025, 3085 ( $\text{CH}=\text{C}$ ), 1075, 1185, 1245, 1730 ( $\text{COO}$ ), 1690 ( $\text{C}=\text{O}$ ).  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm ( $J$ , Hz): 1.13 t (3H,  $\text{OCH}_2\text{CH}_3$ ,  $J = 7.1$ ), 1.16 t (3H,  $\text{OCH}_2\text{CH}_3$ ,  $J = 7.1$ ), 2.02 d (3H,  $=\text{CHCH}_3$ ,  $J = 7.1$ ), 3.39 q (2H,  $\text{OCH}_2\text{CH}_3$ ,  $J = 7.0$ ), 3.43 q (2H,  $\text{OCH}_2$ ), 4.24 s (2H,  $=\text{CCH}_2\text{O}$ ), 6.98 q (1H,  $=\text{CH}$ ,  $J = 7.1$ ).

**Methyl 2-oxo-3-propoxymethyl-3-pentenoate (IVc).** IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 820, 890, 1625, 3025, 3090 ( $\text{CH}=\text{C}$ ), 1075, 1185, 1245, 1735 ( $\text{COO}$ ), 1690 ( $\text{C}=\text{O}$ ).  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm ( $J$ , Hz): 0.91 t (3H,  $\text{CH}_2\text{CH}_3$ ,  $J = 7.4$ ), 1.58 q.t (2H,  $\text{CH}_2\text{CH}_3$ ,  $J = 7.4$ , 6.6), 2.07 d (3H,  $=\text{CHCH}_3$ ,  $J = 7.1$ ), 3.38 t (2H,  $\text{OCH}_2\text{CH}_2\text{CH}_3$ ,  $J = 6.6$ ), 3.88 s (3H,  $\text{OCH}_3$ ), 4.28 s (2H,  $=\text{CCH}_2\text{O}$ ), 7.00 q (1H,  $=\text{CHCH}_3$ ,  $J = 7.1$ ).

**Ethyl 2-oxo-3-propoxymethyl-3-pentenoate (IVd).** IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 820, 890, 1625, 3020, 3085 ( $\text{CH}=\text{C}$ ), 1080, 1180, 1245, 1730 ( $\text{COO}$ ), 1690 ( $\text{C}=\text{O}$ ).  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm ( $J$ , Hz): 0.91 t (3H,  $\text{CH}_2\text{CH}_3$ ,  $J = 7.4$ ), 1.14 t (3H,  $\text{OCH}_2\text{CH}_3$ ,  $J = 7.0$ ), 1.56 q.t (2H,  $\text{CH}_2\text{CH}_3$ ,  $J = 7.4$ , 6.6), 2.08 d (3H,  $=\text{CHCH}_3$ ,  $J = 7.1$ ), 3.39 t (2H,  $\text{OCH}_2\text{CH}_2\text{CH}_3$ ,  $J = 6.6$ ), 3.42 q (2H,  $\text{OCH}_2\text{CH}_3$ ,  $J = 7.1$ ), 4.28 s (2H,  $=\text{CCH}_2\text{O}$ ), 7.02 q (1H,  $=\text{CHCH}_3$ ,  $J = 7.1$ ).

**Methyl (4-vinyl-4-ethoxalyl-1-cyclohexenyl)oxoacetate (VIa).** IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 920, 990, 1630, 1645, 3020, 3090 ( $\text{C}=\text{CH}$ ,  $\text{CH}=\text{CH}_2$ ), 1075, 1140, 1250, 1725 ( $\text{COO}$ ), 1680 ( $\text{C}=\text{O}$ ).  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm ( $J$ , Hz): 2.04 d.d.d (1H,  $\text{CH}_2$ ,  $J = 13.5$ , 7.2, 6.1) and 2.21 d.m (1H,  $\text{CH}_2$ ,  $J = 13.5$ ), 2.30–2.37 m (2H,  $\text{CH}_2$ ), 2.62 d.m (1H,  $\text{CH}_2$ ,  $J = 19.9$ ) and 2.89 d.m (1H,  $\text{CH}_2$ ,  $J = 19.9$ ), 3.83 s (3H,  $\text{OCH}_3$ ), 3.88 s (3H,  $\text{OCH}_3$ ),

5.19 d (1H,  $=\text{CH}_2$ ,  $J = 17.6$ ) and 5.34 d (1H,  $=\text{CH}_2$ ,  $J = 10.7$ ), 5.92 d.d (1H,  $=\text{CH}$ ,  $J = 17.6$ , 10.7), 7.01 t.t (1H,  $=\text{CH}$ ,  $J = 4.1$ , 1.8).  $^{13}\text{C}$  NMR spectrum,  $\delta_{\text{C}}$ , ppm: 19.80 ( $\text{CH}_2$ ), 28.20 ( $\text{CH}_2$ ), 32.50 ( $\text{CH}_2$ ), 51.55 ( $\text{C}$ ), 52.54 ( $\text{OCH}_3$ ), 52.61 ( $\text{OCH}_3$ ), 118.98 ( $=\text{CH}_2$ ), 135.16 ( $=\text{C}$ ), 136.98 ( $=\text{CH}$ ), 145.08 ( $=\text{CH}$ ), 162.33 ( $\text{COO}$ ), 186.48 ( $\text{CO}$ ), 194.77 ( $\text{CO}$ ).

**Ethyl (4-ethoxalyl-4-vinyl-1-cyclohexenyl)oxoacetate (VIb).** IR spectrum,  $\text{cm}^{-1}$ : 920, 990, 1630, 1645, 3025, 3090 ( $\text{C}=\text{CH}$ ,  $\text{CH}=\text{CH}_2$ ), 1070, 1145, 1250, 1725 ( $\text{COO}$ ), 1680 ( $\text{C}=\text{O}$ ).  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm ( $J$ , Hz): 1.34 t (3H,  $\text{OCH}_2\text{CH}_3$ ,  $J = 7.1$ ), 1.37 t (3H,  $\text{OCH}_2\text{CH}_3$ ,  $J = 7.1$ ), 2.03 d.d.d (1H,  $\text{CH}_2$ ,  $J = 13.5$ , 6.1) and 2.19 m (1H,  $\text{CH}_2$ ), 2.30–2.376 m (2H,  $\text{CH}_2$ ), 2.60 d.m (1H,  $\text{CH}_2$ ,  $J = 19.9$ ) and 2.89 d.m (1H,  $\text{CH}_2$ ,  $J = 19.9$ ), 4.28 q (2H,  $\text{OCH}_2\text{CH}_3$ ), 4.33 q (2H,  $\text{OCH}_2\text{CH}_3$ ,  $J = 7.1$ ), 5.18 d (1H,  $=\text{CH}_2$ ,  $J = 17.5$ ) and 5.33 d (1H,  $=\text{CH}_2$ ,  $J = 10.7$ ), 5.92 d.d (1H,  $=\text{CH}$ ,  $J = 17.6$ , 10.7), 6.99 t.t (1H,  $=\text{CH}$ ,  $J = 4.2$ , 1.9).

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