Synthesis and structures of ethoxymethylene derivatives of 1,3-diethoxycyclohexenylium perchlorate

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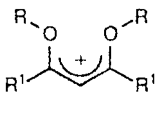
1,3-Diethoxy-4,6-di(ethoxymethylene)-5,5-dimethylcyclohexenylium perchlorate (**6a**) was synthesized from 1,3-diethoxy-5,5-dimethylcyclohexenylium perchlorate by successive introduction of ethoxymethylene groups. Perchlorate **6a** was also obtained from dimedone in one preparative step. Compound **6a** was studied by X-ray diffraction analysis. The bond lengths in the fragment containing the C(1)—C(4) and C(6) atoms of the six-membered ring of the cation are substantially equalized and close to the C_{arom}—C_{arom} bond length. Apparently, this fact indicates that the positive charge is primarily delocalized over the aromatic system.

Key words: 1,3-diethoxy-4-ethoxymethylene-5,5-dimethylcyclohexenylium perchlorate, 1,3-diethoxy-4,6-di(ethoxymethylene)-5,5-dimethylcyclohexenylium perchlorate, 1,3-diethoxy-4,6-di(ethoxymethylene)cyclohexenylium perchlorate, molecular structure, crystal structure, organic synthesis, X-ray diffraction study.

Over a period of the last 10 years, we studied procedures for the preparation and the properties of activated forms of 1,3-dicarbonyl compounds, namely, of 1,3-dihydroxy(dialkoxy)allyl cations (**1**).[1,2] The characteristic feature of these bis-OR-substituted carbocations, which distinguishes them from the initial 1,3-dicarbonyl systems, is deactivation of the central atom of the carbon triad with respect to the attack of an electrophile and, on the contrary, activation of the terminal one or three methyl (methylene) groups of the substituents R^1. For example, condensation of acetylacetone or benzoylacetone with formanilides in the presence of triethyl orthoformate and perchloric acid gave 1-Ar-2-R-4-ethoxypyridinium perchlorates (**2**). Analogously, condensation of acetylacetone with arylaldehydes afforded styrene derivatives, namely, 1,7-diaryl-3,5-diethoxyheptatrienylium perchlorates (**3**).[3] In this work, the above-described condensation reactions were carried out with the use of activated forms of cyclic 1,3-diketones (for example, 1,3-diethoxycyclohexenylium carbocations) with the aim of preparing ethoxymethylene derivatives. The latter are new promising synthons, whose structures combine activated forms of 1,3-diketone and 1,3-ketoaldehyde.



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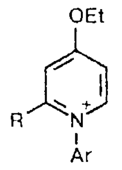

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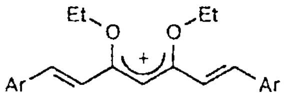

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1,3-Diethoxy-5,5-dimethylcyclohexenylium perchlorate (**4a**) containing activated methylene groups at positions 4 and 6 of the ring has been prepared previously (as a hygroscopic salt) upon *O*-alkylation of dimedone by $(EtO)_3CH$ in the presence of $HClO_4$.[4] However, it appeared that the methylene groups of the above-mentioned cyclic carbocation did not enter into the condensation reaction with arylaldehydes, whereas the methylene groups of the analogous carbocation **4b**, which was prepared from 1,3-cyclohexanedione, reacted with arylaldehydes to form styrene derivatives.[3] This pecu-

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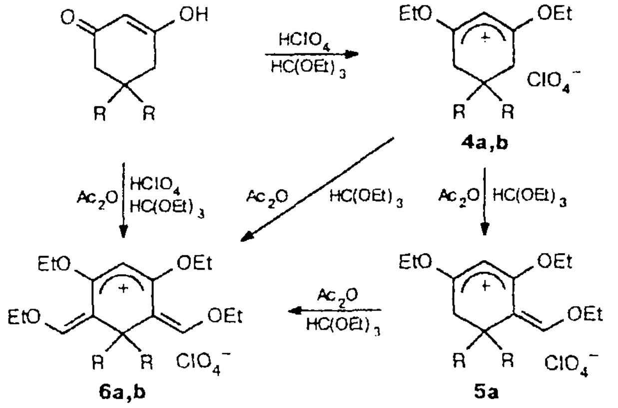
liarity is attributable to spatial shielding of the reactive groups of carbocation **4a** by the methyl substituents at the adjacent carbon atom.



R = Me (**a**), H (**b**)

Previously,[5] it was demonstrated that C—H-active compounds interact with orthoformates in the presence of strong acids to form alkoxymethylene derivatives, which are synthetic equivalents of the aldehyde function. One would expect that the corresponding derivative will be successfully prepared from carbocation **4a** in spite of steric hindrances caused by methyl groups because according to the conventional mechanism of these reactions,[6] the size of the electrophilic particle that attacks the substrate (in our case, the diethoxymethyl carbocation) is substantially smaller than the size of the aryloxymethyl carbocation formed from arylaldehyde.

Actually, we succeeded in introducing successively two ethoxymethylene groups by boiling perchlorate **4a** with $(EtO)_3CH$ in the presence of Ac_2O to prepare the 4-ethoxymethylene (**5a**) and 4,6-di(ethoxymethylene) (**6a**) derivatives, respectively. Perchlorate **6a** can also be obtained from **4a** without isolation of intermediate salt **5a** as well as directly (one-pot procedure) by the reaction of dimedone and an excess of the corresponding orthoether in the presence of Ac_2O and $HClO_4$. In the case of the sterically unshielded 1,3-cyclohexanedione, analogous changes in the reagents and in the conditions of condensation resulted only in bis(ethoxymethylene) derivative **6b**.

The diamagnetic effect of the exocyclic double bonds on the position of the signal from the protons of the 5,5-dimethyl groups can be estimated by comparing the ^{1}H NMR spectra of carbocations **4a**, **5a**, and **6a**. The six-proton singlets from the 5,5-methyl groups of cations **4a**, **5a**, and **6a** are observed at δ 0.95, 1.30, and 1.60, respectively. Therefore, the successive introduction of double bonds leads to the downfield shift of the signal of the methyl groups by approximately δ 0.30—0.35. In addition, solutions of perchlorate of bis(ethoxymethylene) derivative **6a**, unlike the mono derivative **5a**, exhibited green fluorescence, which may be due to the increase in the π-conjugation system and due to the appearance of an axial symmetry in carbocation **6a**.

The final structure of carbocation **6a** in the crystal was established by X-ray diffraction analysis (Figs. 1 and 2; Tables 1—3).

The C(1)...C(6) six-membered ring of the cation adopts a flattened sofa conformation. The C(4) atom deviates from the mean plane through the remaining atoms of the ring by 0.06 Å. The bond lengths in the fragment containing the C(1)—C(4) and C(6) atoms are substantially equalized and close to the C_{arom}—C_{arom} bond length (Table 1). Apparently, the positive charge is primarily delocalized over this system of aromatic bonds of the six-membered ring. Interestingly, the C(4)—C(11) and C(6)—C(16) bonds are virtually not involved in delocalization because their lengths correspond to that of the C=C double bond.

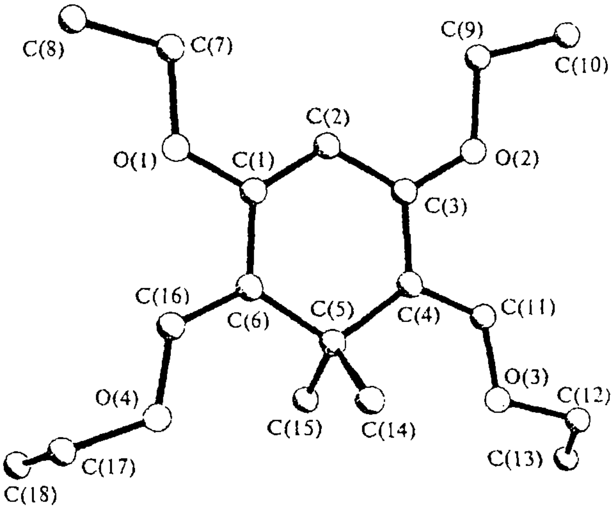


Fig. 1. Structure of cation **6a** (hydrogen atoms are omitted).

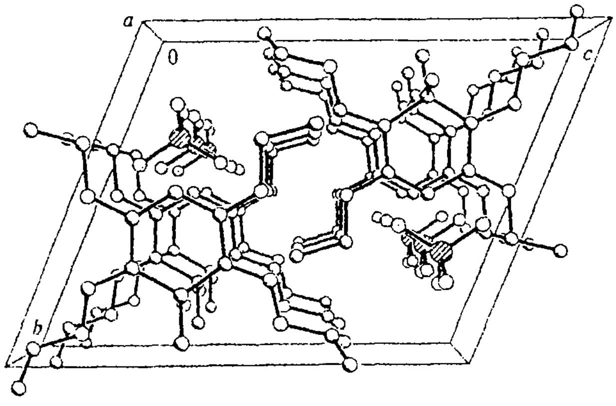


Fig. 2. Crystal structure of compound **6a**.

Table 1. Coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\times 10^3$/ Å^2) of nonhydrogen atoms in the structure of **6a**

Atom	*x*	*y*	*z*	*U*(eq)	Atom	*x*	*y*	*z*	*U*(eq)
Cl(1)	2978(2)	3434(1)	1935(1)	75(1)	C(6)	7095(6)	6842(4)	3993(3)	48(1)
O(1A)	3102(6)	4136(4)	2985(3)	132(2)	C(7)	8143(6)	3603(4)	3755(3)	67(1)
O(2A)	1294(5)	2704(4)	1693(3)	108(1)	C(8)	8084(6)	3219(4)	4654(3)	73(1)
O(3A)	3170(7)	4165(5)	1342(4)	148(2)	C(9)	8031(7)	3676(4)	355(3)	71(1)
O(4A)	4424(5)	2558(4)	1670(2)	97(1)	C(10)	7873(7)	3318(5)	−776(3)	84(2)
O(1)	7740(4)	4936(2)	4144(2)	61(1)	C(11)	7077(6)	7442(4)	1508(3)	70(1)
O(2)	7737(4)	5034(3)	833(2)	66(1)	C(12)	6625(13)	9051(6)	871(4)	143(3)
O(3)	6703(5)	8651(3)	1730(2)	97(1)	C(13)	7746(12)	10018(10)	952(6)	192(4)
O(4)	6348(5)	8459(3)	5563(2)	67(1)	C(14)	4790(6)	7994(4)	3438(3)	75(1)
C(1)	7558(5)	5548(4)	3510(3)	51(1)	C(15)	8132(8)	8841(4)	3774(3)	88(2)
C(2)	7840(5)	4956(4)	2482(3)	58(1)	C(16)	6835(5)	7289(4)	5009(3)	52(1)
C(3)	7578(5)	5602(4)	1844(3)	55(1)	C(17)	6217(6)	8806(4)	6652(3)	64(1)
C(4)	7177(5)	6918(4)	2219(3)	54(1)	C(18)	7947(7)	9462(5)	7188(4)	86(1)
C(5)	6800(6)	7654(4)	3358(3)	56(1)					

Table 2. Bond lengths (*d*) in the structure of **6a**

Bond	*d*/Å	Bond	*d*/Å
Cl(1)—O(1A)	1.389(4)	C(1)—C(6)	1.420(5)
Cl(1)—O(3A)	1.393(4)	C(2)—C(3)	1.384(5)
Cl(1)—O(2A)	1.414(4)	C(3)—C(4)	1.426(5)
Cl(1)—O(4A)	1.438(3)	C(4)—C(11)	1.356(5)
O(1)—C(1)	1.341(4)	C(4)—C(5)	1.535(5)
O(1)—C(7)	1.444(5)	C(5)—C(14)	1.532(6)
O(2)—C(3)	1.331(4)	C(5)—C(15)	1.537(6)
O(2)—C(9)	1.453(5)	C(5)—C(6)	1.539(5)
O(3)—C(11)	1.328(5)	C(6)—C(16)	1.345(5)
O(3)—C(12)	1.455(5)	C(7)—C(8)	1.496(5)
O(4)—C(16)	1.323(4)	C(9)—C(10)	1.493(6)
O(4)—C(17)	1.440(4)	C(12)—C(13)	1.303(8)
C(1)—C(2)	1.368(5)	C(17)—C(18)	1.481(6)

The ethoxy groups at the C(4)—C(11) and C(6)—C(16) double bonds are in *cis* positions with respect to the C(5) atom of the ring. The ethyl groups at the O(3) and O(4) atoms are in antiperiplanar conformations relative to the C(11)—O(3) and C(16)—O(4) bonds, respectively (the C(12)—O(3)—C(11)—C(4) and C(17)—O(4)—C(16)—C(6) torsion angles are −179.2(5)° and −177.0(4)°, respectively). In this case, the shortened H(11)...H(12A) and H(16)...H(17A) intramolecular contacts, whose lengths are 2.09 and 2.18 Å, respectively, are observed (2.32 Å; hereinafter, the sums of the van der Waals radii[7] are given in parentheses).

The ethyl groups at the O(1) and O(2) atoms are in synperiplanar orientations with respect to the C(1)—C(2) and C(2)—C(3) bonds (the C(7)—O(1)—C(1)—C(2) and C(9)—O(2)—C(3)—C(2) torsion angles are −3.5(6)° and 5.6(6)°, respectively) in spite of the shortened intramolecular contacts: O(1)...H(16), 2.19 Å (2.45 Å); C(2)...H(7A), 2.80 Å (2.87 Å); C(2)...H(7B), 2.73 Å; H(2)...C(7), 2.47 Å; H(2)...H(7B), 2.28 Å; O(2)...H(11), 2.20 Å; C(2)...H(9A), 2.70 Å; C(2)...H(9B), 2.83 Å; H(2)...C(9), 2.48 Å; and H(2)...H(9A), 2.27 Å.

The C(13)H$_3$ and C(18)H$_3$ methyl groups are in anticlinal conformations (the C(13)—C(12)—O(3)—C(11) and C(18)—C(17)—O(4)—C(16) torsion angles are −122.8(8)° and 94.5(4)°, respectively). The C(8)H$_3$ and C(10)H$_3$ methyl groups are in antiperiplanar conformations with respect to the corresponding C(sp^3)—O bonds (the C(8)—C(7)—O(1)—C(1) and C(10)—C(9)—O(2)—C(3) torsion angles are −175.2(3)° and 172.0(4)°, respectively).

Table 3. Bond angles (ω) in the structure of **6a**

Angle	ω/deg	Angle	ω/deg	Angle	ω/deg
O(1A)—Cl(1)—O(3A)	114.5(3)	C(2)—C(1)—C(6)	123.7(3)	C(4)—C(5)—C(6)	111.9(3)
O(1A)—Cl(1)—O(2A)	109.0(3)	C(1)—C(2)—C(3)	119.9(4)	C(15)—C(5)—C(6)	108.1(3)
O(3A)—Cl(1)—O(2A)	110.2(3)	O(2)—C(3)—C(2)	121.6(4)	C(16)—C(6)—C(1)	117.0(3)
O(1A)—Cl(1)—O(4A)	107.8(2)	O(2)—C(3)—C(4)	115.5(3)	C(16)—C(6)—C(5)	122.3(3)
O(3A)—Cl(1)—O(4A)	107.4(2)	C(2)—C(3)—C(4)	122.9(3)	C(1)—C(6)—C(5)	120.6(3)
O(2A)—Cl(1)—O(4A)	107.7(2)	C(11)—C(4)—C(3)	116.1(4)	O(1)—C(7)—C(8)	106.2(3)
C(1)—O(1)—C(7)	121.0(3)	C(11)—C(4)—C(5)	123.1(4)	O(2)—C(9)—C(10)	106.6(3)
C(3)—O(2)—C(9)	120.6(3)	C(3)—C(4)—C(5)	120.7(3)	O(3)—C(11)—C(4)	123.5(4)
C(11)—O(3)—C(12)	116.3(4)	C(14)—C(5)—C(4)	107.5(3)	C(13)—C(12)—O(3)	114.3(6)
C(16)—O(4)—C(17)	117.2(3)	C(14)—C(5)—C(15)	112.7(4)	O(4)—C(16)—C(6)	124.2(3)
O(1)—C(1)—C(2)	121.4(4)	C(4)—C(5)—C(15)	108.6(3)	O(4)—C(17)—C(18)	110.7(3)
O(1)—C(1)—C(6)	114.9(3)	C(14)—C(5)—C(6)	108.1(3)		

The $C(14)H_3$ and $C(15)H_3$ methyl substituents at the C(5) atom are in pseudoaxial and pseudoequatorial orientations, respectively (the C(14)—C(5)—C(6)—C(1) and C(15)—C(5)—C(4)—C(3) torsion angles are 115.0(4)° and 124.6(4)°, respectively). This arrangement of the substituents leads to the appearance of shortened intramolecular contacts: O(4)...H(15B), 2.36 Å; O(3)...H(15C), 2.36 Å; C(16)...H(14A), 2.82 Å; C(16)...H(15B), 2.74 Å; C(11)...H(14C), 2.74 Å; and C(11)...H(15C), 2.79 Å.

In the crystals of compound **6a**, the molecules are packed in stacks along the direction (1 0 0) (see Fig. 2). This packing leads to the appearance of the shortened intermolecular contacts: Cl(1)...H(17'A) (1 − *x*, 1 − *y*, 1 − *z*), 3.02 Å (3.06 Å); Cl(1)...H(12'A) (1 − *x*, 1 − *y*, 1 − *z*), 2.97 Å; C(16)...H(8'B) (1 − *x*, 1 − *y*, 1 − *z*), 2.77 Å; and H(10C)...C(11') (1 − *x*, 1 − *y*, 1 − *z*), 2.84 Å.

To summarize, we carried out successive transformations from the propenyl cation to pentadienyl cation **5** and then to heptatrienyl cation **6** starting from one object of investigations, namely, from cation **4**. In this case, ethoxy groups, which additionally stabilize the system as a whole, were introduced simultaneously with an elongation of the carbon chain.

Further synthetic studies of mono- and bis(ethoxymethylene) derivatives **4**, **5**, and **6** are of interest because these compounds are activated forms of 1,3-ketoaldehydes, which are valuable synthons for preparing various heterocyclic compounds, for example, pyrylium and 1-benzopyrylium salts.[8]

Experimental

The IR spectra of samples were recorded on a Specord-71IR spectrometer as Nujol mulls. The ^1H NMR spectra were measured on a Varian VXR-300 spectrometer (300 MHz) in $CDCl_3$.

X-ray diffraction analysis of salt 6a. Crystals of $C_{18}H_{29}O_4{}^+ClO_4{}^-$ are triclinic, at 153(2) K a = 7.314(2) Å, b = 11.361(4) Å, c = 14.154(5) Å, α = 112.75(2)°, β = 90.16(3)°, g = 92.89(3)°, V = 1082.8(6) Å^3, crystal dimensions are 0.5×0.3×0.2 mm, space group $P\bar{1}$, Z = 2, d_{calc} = 1.254 g cm^3, $F(000)$ = 436, μ = 0.215 mm^{-1}.

The intensities of 4150 reflections (3824 independent reflections, R_{int} = 0.055) were measured on an automated four-circle Siemens P3/PC diffractometer (graphite monochromator, Mo-Kα radiation, θ-2θ scanning technique, $2\theta_{max}$ = 50°).

The structure was solved by the direct method with the use of the SHELXTL PLUS program package.[9] The positions of the hydrogen atoms were calculated geometrically and refined using the riding model with fixed values of $U_{iso} = nU_{eq}$ of the nonhydrogen atoms attached to the hydrogen atoms (n = 1.5 for the methyl groups and n = 1.2 for the remaining hydrogen atoms). The refinement was carried out based on F^2 by the full-matrix least-squares method with anisotropic thermal parameters for nonhydrogen atoms (250 parameters) using 2249 reflections. The refinement converged to R_1 = 0.058 (based on 1615 reflections with $F > 4\sigma(F)$), wR_2 = 0.155, S = 1.05. The coordinates of nonhydrogen atoms are given in Table 1. The bonds lengths and bond angles are listed in Tables 2 and 3, respectively.

1,3-Diethoxy-4-ethoxymethylene-5,5-dimethylcyclohexenylium perchlorate (5a). A mixture of 1,3-diethoxy-5,5-dimethylcyclohexenylium perchlorate (**4a**)[4] (3.0 g, 0.01 mol), Ac_2O (1.2 mL), and $(EtO)_3CH$ (4.3 mL) was heated to weak reflux and boiled to distill off low-boiling fractions over 40 min. Then orthoether (0.6 mL) was added. The reaction mixture was boiled for 30 min, cooled with ice water, and diluted with Et_2O until the mixture became turbid. Crystallization was initiated by rubbing. The mixture was kept for 1 h to obtain crystals. The product was filtered off and washed with a 4 : 1 Et_2O—AcOH mixture and then with Et_2O. Compound **5** was obtained in a yield of 2.1 g (59%) as yellow crystals, m.p. 93—94 °C (from Ac_2O). ^1H NMR, δ: 1.30 (s, 6 H, 5-$C(CH_3)_2$ + 4-(=$CHCH_2\underline{CH}_3$); 1.38—1.55 (m, 9 H, 1,3-$(OCH_2\underline{CH}_3)_2$ + 4-(=$CHCH_2\underline{CH}_3$)); 2.53 (s, 2 H, CH_2); 4.38 (q, 2 H, 1-$O\underline{CH}_2CH_3$, ^3J = 7 Hz); 4.50—4.67 (m, 4 H, 3-$O\underline{CH}_2CH_3$ + 4-(=$CH-O\underline{CH}_2CH_3$); 6.22 (s, 1 H, 2-H); 7.94 (s, 1 H, 4-(=$C\underline{H}-OCH_2CH_3$). IR, ν/cm^{-1}: 1600, 1580, 1260, 1200, 1100. Found (%): C, 51.04; H, 7.12; Cl, 10.01. $C_{15}H_{25}ClO_7$. Calculated (%): C, 51.07; H, 7.14; Cl, 10.05.

1,3-Diethoxy-4,6-di(ethoxymethylene)-5,5-dimethylcyclohexenylium perchlorate (6a). *A.* A mixture of 1,3-diethoxy-5,5-dimethylcyclohexenylium perchlorate (**4a**) (2.96 g, 0.01 mol), $(EtO)_3CH$ (8 mL), and Ac_2O (20 mL) was heated to weak reflux. The mixture was boiled to distill off low-boiling fractions over 40 min. Then orthoether (5.5 mL) was added. The reaction mixture was boiled for 40 min, cooled with water, and diluted with a 3—4-fold volume of Et_2O. Crystallization was initiated by rubbing. The mixture was kept for 2 h to obtain crystals. The product was filtered off and washed with Et_2O. Compound **6a** was obtained as yellow-brown crystals in a yield of 2.7 g (75%), m.p. 116—117 °C (from Ac_2O). ^1H NMR, δ: 1.40 (t, 6 H, 1,3-$(OCH_2\underline{CH}_3)_2$, ^3J = 7 Hz); 1.48 (t, 6 H, 4,6-(=$CH-OCH_2\underline{CH}_3)_2$, ^3J = 7 Hz); 1.60 (s, 6 H, 5-$C(CH)_3)_2$); 4.29 (q, 4 H, 1,3-$(O\underline{CH}_2CH_3)_2$, ^3J = 7 Hz); 4.51 (q, 4 H, 4,6-(=$CH-O\underline{CH}_2CH_3)_2$, ^3J = 7 Hz); 6.00 (s, 1 H, 2-H); 7.63 (s, 2 H, 4,6-(=$\underline{CH}-OCH_2CH_3)_2$). IR, ν/cm^{-1}: 1600, 1580, 1260, 1200, 1100. Found (%): C, 52.83; H, 7.14; Cl, 8.64. $C_{18}H_{29}ClO_8$. Calculated (%): C, 52.88; H, 7.15; Cl, 8.67.

B. A mixture of salt **5** (3.5 g, 0.01 mol), $(EtO)_3CH$ (10 mL), and Ac_2O (20 mL) was boiled to distill off low-boiling fractions over 1 h. The reaction mixture was cooled with an equal volume of Et_2O and kept for 2 h to obtain crystals. The yield was 3.4 g (85%).

C. A mixture of dimedone (1.4 g, 0.01 mol), $(EtO)_3CH$ (30 mL), and a 70% $HClO_4$ solution (1 mL) was heated to boiling. Then Ac_2O (30 mL) was added and the reaction mixture was boiled for ~2 ч to distill off low-boiling fractions until the temperature of the vapor that removed reached 100—120 °C. The mixture was cooled and diluted with an equal volume of Et_2O. The mixture was kept to obtain crystals. The yield was 3.5 g (86%).

1,3-Diethoxy-4,6-di(ethoxymethylene)cyclohexenylium perchlorate (6b) was prepared according to the above-described procedure from cyclohexane-1,3-dione (1.1 g 0.01 mol) as yellow crystals in a yield of 1.6 g (42%), m.p. 158—160 °C (from Ac_2O). Crystals were kept in a desiccator over P_2O_5. ^1H NMR, δ: 1.38 (t, 6 H, 1,3-$(OCH_2\underline{CH}_3)_2$); 1.48 (t, 6 H, 4,6-(=$CH-OCH_2\underline{CH}_3)_2$, ^3J = 7 Hz); 3.35 (s, 2 H, CH_2); 4.30 (q, 4 H, 1,3-$(O\underline{CH}_2CH_3)_2$, ^3J = 7 Hz); 4.53 (q, 4 H, 4,6-(=$CH-O\underline{CH}_2CH_3)_2$, ^3J = 7 Hz); 6.00 (s, 1 H, 2-H); 7.63 (s, 1 H, 4,6-(=$\underline{CH}-OCH_2CH_3)_2$). IR, ν/cm^{-1}: 1600, 1580, 1260, 1200, 1100. Found (%): C, 50.43; H, 6.59; Cl, 9.31. $C_{16}H_{25}ClO_8$. Calculated (%): C, 50.46; H, 6.62; Cl, 9.31.

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