1), 7.48–7.40 (m, 3), 7.21 (d, J = 6.6 Hz, 1), 3.66 (center of AA'XX' system, 4 peri-bridge H).

1-Aceanthrenone (4). Aluminum chloride (8 g, 0.06 mol) was added over a period of 45 min to a solution of anthracene (5.35 g, 0.03 mol) and chloroacetyl chloride (2.4 mL, 0.03 mol) in methylene chloride at −5 to 0 °C. The dark colored reaction mixture was kept at room temperature for 24 h and then decomposed with ice–hydrochloric acid. The organic layer was subsequently washed with sodium bicarbonate and water and dried over $MgSO_4$. The dark colored solution thus obtained was treated twice with charcoal, and part of the solvent was removed by vacuum evaporation to give 300–400 mg of an insoluble yellow crystalline precipitate which consists of a mixture of disubstituted chloroacetylanthracenes (not further investigated). The filtrate was subjected to flash chromatography (SiO_2/CH_2Cl_2), and the yellow crystalline product (R_f 0.33) was first sublimed (110 °C (0.03 mm)) and then recrystallized from methylene chloride by addition of cyclohexane: yield 2.48 g (38%) of yellow crystals; mp 157–158 °C (lit.[9] mp 151–152 °C); IR (KBr) 1680 cm^{-1} (C=O); ^1H NMR ($CDCl_3$) δ 9.18 (d, J = 8.3 Hz, 1), 8.64 (s, 1), 8.14 (d, J = 7.9 Hz, 1), 7.91 (d, J = 8.6 Hz, 1), 7.77–7.71 (m, 1), 7.64–7.52 (m, 2), 7.42 (d, J = 6.7 Hz, 1), 3.90 (s, 2). Anal. Calcd for $C_{16}H_{10}O$: C, 88.05; H, 4.62. Found: C, 88.19; H, 4.56.

1-Aceanthrenol (5). Sodium borohydride (0.8 g) was added to a solution of 1-aceanthrenone (1 g, 4.6 mmol) in a mixture of methylene chloride (50 mL) and methanol (50 mL). The reaction mixture was refluxed for 15 min and then neutralized with aqueous acetic acid (50%). The methylene chloride was removed from the organic layer by vacuum evaporation, and the remaining solution was diluted with water (50 mL) to give a crystalline precipitate. It was removed by filtration and recrystallized from methylene chloride by adding hexane: yield 0.88 g (87%) of yellow, needle-shaped crystals; mp (dependent on the rate of heating) 175–185 °C (partly dec); ^1H NMR ($CDCl_3$) δ 8.36 ("d", J = 10.6 Hz, 1), 8.33 (s, 1), 8.08 ("d", J = 9.7 Hz, 1), 7.76 (d, J = 8.6 Hz, 1), 7.57–7.43 (m, 3), 7.26–7.23 (m, 1), 6.24 (dd, J = 7.8, 6.4 Hz, 1), 3.94 (dd, J = 18.0, 6.4 Hz, 1), 3.38 (d, J = 18.0 Hz, 1), 2.04 (d, J = 7.8 Hz, 1 OH). Anal. Calcd for $C_{16}H_{12}O$: C, 87.24; H, 5.49. Found: C, 87.04; H, 5.41.

Aceanthrenequinone (6) was prepared according to ref 10.

1,2-Aceanthrenediols (7/8). Sodium borohydride (2.32 g, 61 mmol) was added under nitrogen to a suspension of aceanthrenequinone (2.32 g, 10 mmol) in a mixture of methylene chloride (350 mL) and 95% ethanol (70 mL) placed in a separatory funnel. The reaction mixture was agitated by a gentle stream of nitrogen for 2 h until a light yellow solution had formed. The solution was then extracted once with 100 mL of water, and neutralization of this aqueous solution with acetic acid afforded 100 mg of virtually pure cis diol 7 (vide infra). The organic layer was washed 3 times with 100-mL portions of water whereupon part of the diol mixture precipitated from the methylene chloride solution. Partial removal of solvent by vacuum evaporation, followed by addition of hexane, gave 1.96 g of yellow crystalline cis/trans diol mixture. Separation into cis and trans isomers was accomplished by fractional crystallization from boiling ethanol in which the cis diol 7 (550 mg) is less soluble. It forms bright yellow needles, mp 239–242 °C. The yellow mother liquor was treated briefly with little sodium borohydride, and conventional workup afforded the trans diol 8 (1.10 g). It is rather soluble in acetone and can be precipitated with water. It forms almost colorless, fluffy needle-shaped crystals, mp 185–187 °C. Spectroscopic and analytical data of 7 and 8 as follow.

***cis*-1,2-Aceanthrenediol (7):** ^1H NMR ($CDCl_3$) δ 8.42 ("d", J = 8.1 Hz, 1), 8.38 (s, 1), 8.10 ("d", J = 8.1 Hz, 1), 7.91–7.88 (m, 1), 7.64–7.52 (m, 4), 6.01 (dd, J = 7.0, 6.0 Hz, 1), 5.65 (dd, J = 7.0, 6.0 Hz, 1), 2.84 (d, J = 7.0 Hz, 1 OH), 2.83 (d, J = 7.0 Hz, 1 OH). Anal. Calcd for $C_{16}H_{12}O_2$: C, 81.34; H, 5.12. Found: C, 81.16; H, 5.04.

***trans*-1,2-Aceanthrenediol (8):** ^1H NMR ($CDCl_3$) δ 8.39 (s, 1), 8.38 ("d", J = 7.3 Hz, 1), 8.10 ("d", J = 7.5 Hz, 1), 7.91 ("d", J = 8.3 Hz, 1), 7.59–7.49 (m, 4), 5.97 (d, J = 7.5 Hz, 1), 5.59 (d, J = 6.8 Hz, 1), 2.31 (d, J = 7.5 Hz, 1 OH), 2.24 (d, J = 6.8 Hz, 1 OH). Anal. Calcd for $C_{16}H_{12}O_2$: C, 81.34; H, 5.12. Found: C, 81.06; H, 5.01.

2-Aceanthrenone (9). A. From Cis Diol 7. *p*-Toluenesulfonic acid (15 mg) was added to a hot suspension of 7 (354 mg, 1.5 mmol) in toluene (15 mL), and the dark brown reaction mixture was refluxed for 1 h. Workup by extraction with sodium bicarbonate, washing, and drying, followed by flash chromatography on SiO_2/CH_2Cl_2 and vacuum sublimation (115 °C (0.04 mm)), gave 269 mg (82%) of yellow crystals, mp 167 °C. The substance may be recrystallized from methylene chloride by precipitation with hexane: ^1H NMR ($CDCl_3$, 6 mg/mL, concentration-dependent) δ 8.45 (s, 1), 8.24 (d, J = 8.4 Hz, 1), 8.12–8.09 (m, 1), 8.00–7.97 (m, 2), 7.75–7.69 (m, 1), 7.58–7.53 (m, 2), 4.16 (d, J = 0.8 Hz, 2). Irradiation of the multiplet centered at δ 7.98 changes the doublet at δ 4.16 into a singlet: IR (KBr) 1700 cm^{-1}. Anal. Calcd for $C_{16}H_{10}O$: C, 88.05; H, 4.62. Found: 87.86; H, 4.57.

B. From Trans Diol 8. The reaction was carried out as described above for diol 7 and gave 214 mg (65%) of 9.

C. From Crude Cis,Trans Diol Mixture. A suspension of crude 7/8 (2.8 g) and *p*-toluenesulfonic acid (100 mg) in toluene (100 mL) was refluxed for 1 h. Workup as described under A gave 1.5 g (59%) of 9.

2-Aceanthrenol (10). The reduction of 2-aceanthrenone was carried out in the same fashion as described for 1-aceanthrenone: yield 91% of yellow, needle-shaped crystals (from methylene chloride/hexane); mp 209–210 °C; ^1H NMR ($CDCl_3$) δ 8.26 (s, 1), 8.08–7.96 (m, 2), 7.88 (d, J = 7.8 Hz, 1), 7.58–7.47 (m, 4), 5.89 (dd, J = 7.0, 7.5 Hz, 1), 4.16 (dd, J = 7.0, 18.0 Hz, 1), 3.62 (d, J = 18.0 Hz, 1), 2.04 (d, J = 7.5 Hz, 1 OH). Anal. Calcd for $C_{16}H_{12}O$: C, 87.24; H, 5.49. Found: C, 87.18; H, 5.37.

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Registry No. 2, 202-03-9; 3, 641-48-5; 4, 51752-51-3; 5, 93645-78-4; 6, 6373-11-1; *cis*-7, 90047-28-2; *trans*-8, 90047-31-7; 9, 90047-29-3; 10, 90047-30-6; anthracene, 120-12-7.

Stereoselective Synthesis of (±)-11-Hydroxy-*trans*-8-dodecenoic Acid from 10-Undecenoic Acid

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In a previous paper,[1] we reported a versatile method for the stereoselective synthesis of β,γ-unsaturated ketones by reductive deconjugation of α-bromo α,β-unsaturated ketones which are readily prepared by the treatment of silyl enol ethers with dibromocarbene. This process achieves the selective introduction of a carbon–carbon double bond β,γ to ketones with one-carbon homologation. Now we describe an effective synthetic route to (±)-11-hydroxy-*trans*-8-dodecenoic acid (1), a precursor of (±)-recifeiolide (2),[2,3] from the commercially available 10-un-

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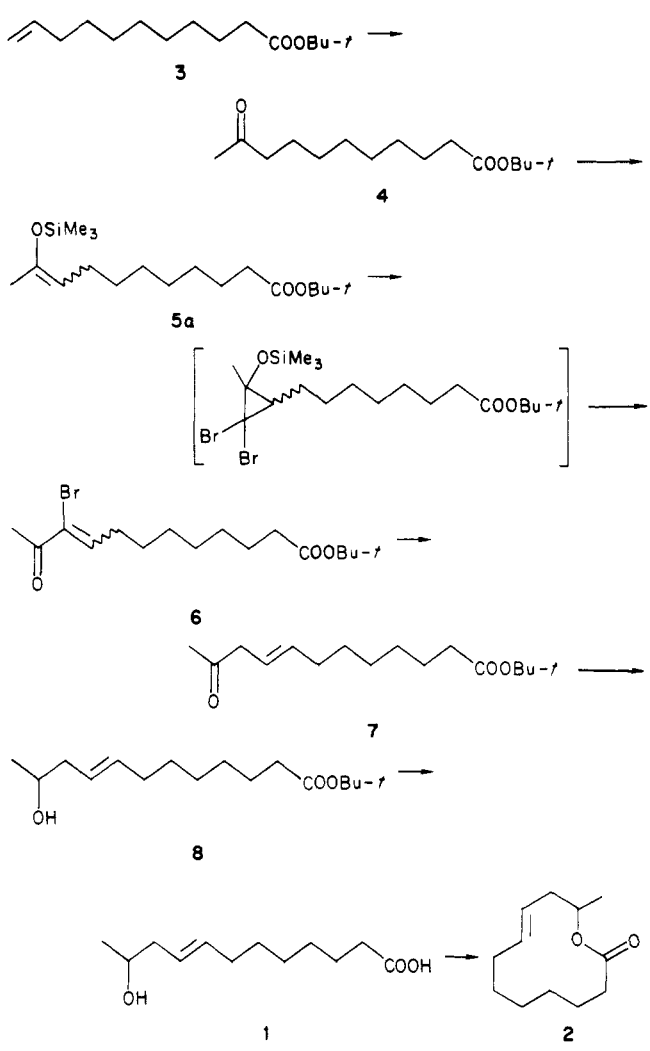
Scheme I



decenoic acid by using the present methodology as a key step (Scheme I).

The *tert*-butyl ester **3** of 10-undecenoic acid underwent Wacker's oxidation with $PdCl_2/CuCl/H_2O/O_2$ in DMF to produce the keto ester **4** in 93% yield. Reaction of **4** with chlorotrimethylsilane and triethylamine in DMF gave the silyl enol ether **5a** and *tert*-butyl 10-(trimethylsiloxy)-10-undecenoate (**5b**) as the regioisomer in 62% (**5a**:**5b** 81:19) yield. The mixture was subjected to the addition of dibromocarbene, followed by ring opening with diethyl phosphonate (commercially named diethyl phosphite). The desired α-bromo α,β-unsaturated ketone **6** was easily separated by flash chromatography in 43% yield based on **5**.[4] Use of the ethyl ester instead of the *tert*-butyl derivative **5a** led to a poor yield of the corresponding α-bromo α,β-unsaturated ketone under the conditions employed here.

Reductive deconjugation of **6** was accomplished by treatment with diethyl phosphonate and triethylamine and resulted in the stereoselective formation of the *trans*-β,γ-unsaturated ketone **7** (77%) with no detectable production of the cis isomer or α,β-unsaturated ketone. This high selectivity is of the key features of the present approach.

The ketone **7** was reduced to the alcohol **8** with $NaBH_4$. Hyrolysis of the *tert*-butyl ester **8** with chlorotrimethylsilane and NaI in acetonitrile gave the desired hydroxy acid **1** (87% yield based on **7**) which was confirmed by comparison of spectral data with reported values.[3a-d] This hydroxy acid **1** is known to cyclize into (±)-recifeiolide (**2**).

Experimental Section

***tert*-Butyl 10-Oxo-10-undecenoate (4).** Oxygen (40 mL/min) was bubbled into a solution of $PdCl_2$ (1.01 g, 5.7 mmol), CuCl (5.44 g, 55 mmol) and H_2O (3.4 mL) in DMF (29 mL) for 1 h, and then *tert*-butyl 10-undecenoate (**3**, 13.0 g, 54 mmol) was added at room temperature.[5] Bubbling of oxygen was continued for 6 h with stirring at room temperature. After the addition of 3 N HCl (30 mL), the resultant mixture was extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layers were washed with water (1 × 50 mL) and then 10% K_2CO_3 (aqueous) (1 × 50 mL), dried over $MgSO_4$, and concentrated. The keto ester **4**[6] (12.9 g, 93%) was obtained by conventional distillation (82–87 °C (0.05–0.07 mmHg)). **4**: IR (neat) 1720, 1710 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ 0.8–2.6 (m, 16 H), 1.40 (s, 9 H), 2.07 (s, 3 H).

***tert*-Butyl 10-Bromo-11-oxo-9-dodecenoate (6).** The silyl enol ether **5** was prepared in 62% yield (**5a**:**5b** 81:19; determined by ^1H NMR) by the reaction of **4** with chlorotrimethylsilane and triethylamine in DMF at 90 °C for 30 h according to the reported method.[7] To a suspension of potassium *tert*-butoxide (0.75 g, 6.7 mmol) in hexane (3.6 mL) and benzene (2.4 mL) was added **5** (1.08 g, 3.3 mmol) at −20 to −15 °C, followed by dropwise addition of bromoform (1.16 g, 4.6 mmol) over 45 min at the same temperature. Stirring was continued for 0.5 h, and then the reaction temperature was raised to room temperature. After further stirring for 5 h, 20 mL of water was added to the resultant mixture which was extracted with ether (3 × 30 mL). The combined organic layers were washed with brine, dried over $MgSO_4$, and concentrated. The residue was treated with diethyl phosphonate (1.38 g, 10 mmol) at 50 °C for 1 h. This mixture was flash chromatographed, eluting with 10% ether–hexane to give 0.495 g (43% based on **5**) of **6** (the geometry was not determined). **6**: TLC R_f 0.46 (10% EtOAc–benzene); IR (neat) 1725, 1686, 1604 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 0.8–2.6 (m, 14 H), 1.40 (s, 9 H), 2.37 (s, 3 H), 7.01 (t, 1 H, J = 7.0 Hz); MS, m/e 348 (M^+).

***tert*-Butyl 11-Oxo-*trans*-8-dodecenoate (7).** A mixture of diethyl phosphonate (0.197 g, 1.43 mmol), triethylamine (0.579 g, 5.72 mmol), and **6** (0.495 g, 1.43 mmol) was heated at 80 °C for 2.5 h. The separated white deposit ($Et_3N{\cdot}HBr$) was filtered off and washed with 10 mL of ether. The filtrate and ethereal washings were concentrated and flash chromatographed, eluting with 1:10 ether–hexane to give 0.296 g (77%) of **7** which was isomeric pure by NMR and TLC (R_f 0.38, 10% EtOAc–benzene). **7**: IR (neat) 1723, 960 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 0.8–2.6 (m, 12 H), 1.42 (s, 9 H), 2.11 (s, 3 H), 2.9–3.2 (m, 2 H), 5.1–5.8 (m, 2 H); ^{13}C NMR (23 MHz, $CDCl_3$) δ 206.4, 172.7, 134.8, 121.9, 79.5, 47.4, 35.3, 32.3, 28.9, 28.7, 28.5, 27.9, 24.9; MS, m/e 212 ($M^+ - C_4H_8$). Anal. Calcd for $C_{16}H_{28}O_3$: C, 71.60; H, 10.52. Found: C, 71.42; H, 10.70.

(±)-11-Hydroxy-*trans*-8-dodecenoic Acid (1). The ester **7** (0.102 g, 0.38 mmol) was treated with $NaBH_4$ (10 mg, 0.27 mmol) in ethanol (10 mL) for 1 h. Acetic acid (1 or 2 drops) was added to the resultant mixture. After stirring for 10 min, 20 mL of water was added to the mixture which was extracted with ether (3 × 20 mL). The aqueous layer was adjusted to pH 2 with 3 N HCl and extracted with ether (3 × 10 mL). The combined organic layers were washed with brine, dried over $MgSO_4$, and concentrated to give 94.5 mg of the product **8** which was pure by ^1H NMR and TLC (R_f 0.36, 20% EtOAc–benzene). **8**: IR (neat) 3400, 1728, 968 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 1.0–2.2 (m, 15 H), 1.16 (d, 3 H, J = 6.2 Hz), 1.43 (s, 9 H), 3.70 (sextet, 1 H, J = 6.2 Hz), 5.1–5.6 (m, 2 H); MS, m/e 226 ($M^+ - C_2H_4O$).

To a mixture of this product (9.9 mg, theoretically 0.037 mmol) and dry NaI (9.1 mg, 0.061 mmol) in acetonitrile (0.3 mL) were added chlorotrimethylsilane (6.1 mg, 0.056 mmol) and pyridine (0.3 μL, 0.004 mmol) under nitrogen.[8] The mixture was stirred

(4) The isomer **5b** was converted to *tert*-butyl 11-bromo-10-oxo-11-dodecenoate in the process **5** → **6**.

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for 1 h at room temperature, and after the addition of water (0.1 mL), the resultant mixture was stirred for 10 min. Ether (20 mL) was added to the mixture which was washed with 3% $Na_2S_2O_3$ (aqueous) (0.15 mL) and then brine, dried over $MgSO_4$, and concentrated to give 7.5 mg of a colorless oil 1, pure by ^1H NMR and TLC (R_f 0.24, 20% EtOAc–benzene). The yield was 87% based on 7. 1: IR (neat) 3400, 1710, 968 cm^{-1}; ^1H NMR (90 MHz, $CDCl_3$) δ 1.1–2.5 (m, 12 H), 1.19 (d, 3 H, J = 6.2 Hz), 2.34 (t, 2 H, J = 7.1 Hz), 3.2–4.4 (m, 2 H), 3.79 (sextet, 1 H, J = 6.2 Hz), 5.1–5.7 (m, 2 H).

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Registry No. 1, 58654-19-6; 2, 64418-66-2; 3, 93757-41-6; 4, 35005-46-0; (*E*)-5, 93757-42-7; (*Z*)-5, 93757-43-8; 5 ethyl ester deriv, 93757-44-9; 6, 93757-45-0; 6 ethyl ester deriv, 93757-46-1; 7, 93757-47-2; 8, 93757-48-3; *tert*-butyl 11-bromo-10-oxo-11-dodecenoate, 93781-83-0.

Communications

Autoxidation of Micelle-Solubilized Linoleic Acid. Relative Inhibitory Efficiencies of Ascorbate and Ascorbyl Palmitate

Summary: The autoxidation of linoleic acid in sodium dodecyl sulfate micelles is inhibited by water-soluble ascorbate and, to a much greater extent, by lipid-soluble ascorbyl palmitate.

Sir: The autoxidation of membrane-bound polyunsaturated fatty acids occurs by a free radical mechanism to yield lipid hydroperoxides as primary products.[1] Despite the known susceptibility of membrane lipids to oxidation, relatively few quantitative studies on the autoxidation of biological molecules in model membranes have been reported. Pioneering studies by Barclay and Ingold have demonstrated that the same rate law describes the autoxidation of egg lecithin in both homogeneous solution and as multilamellar liposomes.[2] More recent studies have described facets of the autoxidation of linoleic acid in sodium dodecyl sulfate (SDS) micelles[3,4] and of dilinoleoylphosphatidylcholine liposomes.[4] Despite the segregation of reagents provided by the micelles and liposomes, these studies have shown that water-soluble antioxidants can inhibit the autoxidation of lipophilic substrates. This result has important implications for biological systems since several naturally occurring hydrophilic antioxidants have been suggested to play a role in protecting cell membranes from lipid peroxidation in vivo.[5] For this reason, we have undertaken a study of the relative efficiencies of water-soluble ascorbate (1) and lipid-soluble ascorbyl palmitate (2) in inhibiting the autoxidation of micelle-solubilized linoleic acid. Since no significant difference in intrinsic reactivity is expected for two such structurally similar compounds, a comparison of the antioxidant efficiencies of 1 and 2 should provide a useful probe of effects due to the biphasic medium.

The system under study is similar to that described by Barclay et al.[3] The autoxidation of linoleic acid (6.90 mM) in SDS/phosphate buffer (50 mM each, pH 7.0) was initiated by using the lipophilic azo compound di-*tert*-butyl hyponitrite[6] (DBHN, 0.306 mM). As shown in Figure 1,

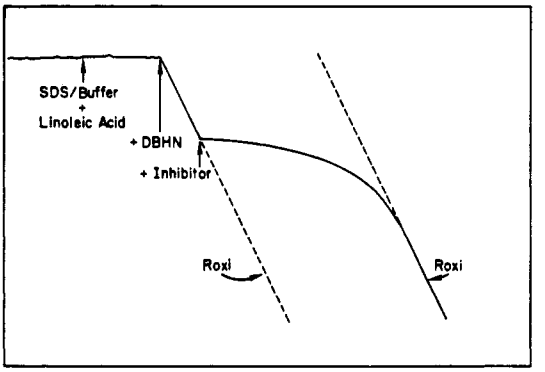

Figure 1. Typical oxygen electrode trace for the autoxidation of linoleic acid (6.90 mM) in SDS micelles at 40.0 °C. The initiator (DBHN, 0.306 mM) and inhibitor (2, 1.48 μM) were added at the points shown on the trace. Note that the rate of oxygen consumption (R_{oxi}) returns to its original value after the added inhibitor is consumed.

Table I. Data for the Inhibited Autoxidation of Linoleic Acid in SDS Micelles at 40.0 °C

ascorbate (1)		ascorbyl palmitate (2)	
1 (μM)	$-d(O_2)/dt$ $\times 10^9$ (M s^{-1})	2 (μM)	$-d(O_2)/dt$ $\times 10^9$ (M s^{-1})
0	82.4	0	77.4
46.0	12.8	1.02	8.57
69.0	9.96	1.63	6.48
115	7.80	2.45	5.50
195	6.11	3.67	4.30
333	5.18	5.71	3.78
564	4.80	7.75	3.39

the rate of oxidation is quite slow in the absence of added initiator but proceeds at a constant and rapid rate immediately upon addition of the DBHN.[7] Similarly, the addition of either lipophilic or hydrophilic antioxidants has an immediate inhibitory effect on the reaction. This reflects the fact that diffusion into the micelle is known to be a fast process,[8] so that initiators and inhibitors can be added to the bulk aqueous phase and will equilibrate into the micelle too fast for any delay to be observed in the kinetic traces.

The rate of oxidation of micelle-solubilized linoleic acid was studied as a function of added inhibitor concentration

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