and heated at 100° for 48 hr in a steel container. The dark yellow reaction mixture was concentrated to dryness and the residue was mixed with water (2 ml) and decolorized with charcoal Darco 60. The colorless aqueous solution was evaporated to dryness and coevaporated several times with ethanol. The residue was triturated twice with chloroform (2 ml each) and the residue was finally crystallized from water–ethanol to give colorless needles: 27.4 mg (60%); mp ~235° (browning), 253–256° (effervesced).

**$N^4$-Acetyl-1-(4-*O*-acetyl-2,3-dideoxy-α-L-*glycero*-pentopyranosyl)cytosine (17).** A mixture of 1 (17 mg), acetic anhydride (0.5 ml), and pyridine (2 ml) was kept at room temperature for 16 hr. The reaction was stopped by addition of water (5 ml) followed by extraction with chloroform (2 × 5 ml). The organic extracts were washed with 5 ml each of water, saturated sodium bicarbonate solution, and water and dried over sodium sulfate. After removal of the solvent, traces of pyridine were removed by coevaporation of ethanol. The crystalline residue was recrystallized from ethanol, 7 mg, mp 200–202°, unchanged on admixture with an authentic sample.[2]

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**Registry No.**—**1**, 39057-02-8; **3**, 51838-76-7; **4**, 51820-58-7; **5**, 51820-59-8; **6**, 51820-60-1; **7**, 51820-61-2; **8**, 51820-62-3; **9**, 51820-63-4; **10**, 51820-64-5; **11**, 51838-77-8; **12**, 51820-65-6; **13**, 51838-78-9; **14**, 51820-66-7; **15**, 51820-67-8; **16**, 51820-68-9; **17**, 51820-69-0; $N^4$-anisoylcytosine, 51820-70-3; tri-*O*-acetyl-L-arabinopyranosyl bromide, 51830-02-5.

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# Electrolytic Decarboxylation Reactions. I. Electrosyntheses of γ-Substituted Butyrolactones and γ-Substituted α,β-Butenolides from γ-Substituted Paraconic Acids

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The product-selective electrolytic decarboxylation of γ-substituted paraconic acids has been studied (1) in dry methanol using sodium methoxide by addition of iron powder or ferric nitrate on platinum electrodes, (2) in dry methanol using sodium methoxide on carbon rod electrodes, and (3) in a mixed solvent of triethylamine–pyridine–water on carbon rod electrodes. Conditions 1 and 2 resulted in exclusive formation of γ-substituted butyrolactones in 80–99% yields, whereas condition 3 provided α,β-unsaturated butenolides in 70–90% yields. By means of the butenolide synthesis *dl*-3-carboxy-8-hydroxy-$\Delta^3$-menthene γ-lactone, a key intermediate for the preparation of *dl*-menthone, could be prepared.

The value of non-Kolbe type electrolytic reactions for the preparation of synthetic intermediates has been discussed recently.[1] Choices of electrodes, solvents, supporting electrolytes, additives, etc., in relation to product selectivity have been the subject of several investigations.[2] We report herein the product-selective electrolytic decarboxylation reaction of γ-substituted paraconic acids (1), which led to the discovery of a chemically controlled electrolysis.

Preliminary electrolysis[3] of **1** [$R_1$, $R_2$ = $-(CH_2)_5-$][4] in dry methanol using sodium methoxide as a supporting electrolyte on platinum electrodes (Table I, run 1) afforded lactone derivatives of **2** (32%), **3** (49%), and **4** (10%). However, addition of iron powder or ferric nitrate in the electrolytic

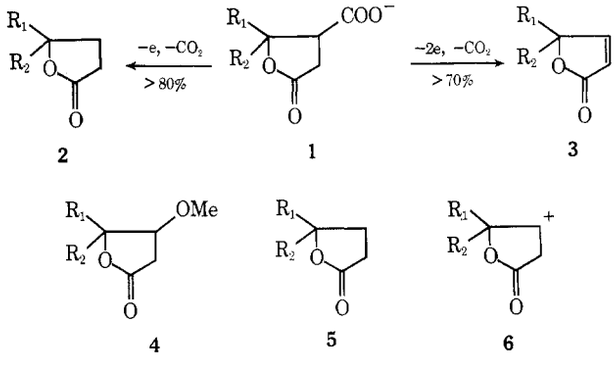


**Table I**
**Electrolytic Conditions for the Anodic Oxidation of γ,γ-Pentamethyleneparaconic Acid (300 mg)**

| Run | Electrode | Supporting electrolyte (mg) | Solvent (ml) | Current, A | Applied voltage, V | Temp, °C | Time, hr | Product, %[a] 2 | 3 | 4 |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Pt | MeONa (940) | MeOH (40) | 1.25 | 10–17 | 20 | 12 | 32 | 49 | 10 |
| 2 | Pt | MeONa–Fe (940:100) | MeOH (40) | 1.25 | 10–17 | 20 | 12 | 80 | | |
| 3 | C | MeONa (940) | MeOH (40) | 1.5 | 7–15 | 25–35 | 20 | 99 | | |
| 4 | C | $Et_3N$ (30) | Py-$H_2O$ (30:4) | 0.25–0.1 | 60–80 | 20 | 12 | | 78 | |

[a] Yields are calculated on isolated product.

**Table II**
**Electrosynthesis of γ-Butyrolactones (2)**

| Product, butyrolactones (2) $R_1$ | $R_2$ | Registry no. | Results, % yield[a] MeOH–MeONa–Fe–(Pt) (run 2) | MeOH–MeONa–(C) (run 3) | Ref |
|---|---|---|---|---|---|
| Me | Me | 3123-97-5 | 97 | 92 | *b* |
| Me | $n$-$C_3H_7$ | 3284-93-3 | 99 | 94 | *b* |
| Me | $n$-$C_6H_{13}$ | 7011-83-8 | 99 | 95 | *c* |
| $-(CH_2)_4-$ | | 33448-80-5 | 81 | 98 | *d* |
| $-(CH_2)_5-$ | | 699-61-6 | 80 | 99 | *e* |

[a] Yields are calculated on isolated product. [b] S. Dev and C. Rai, *J. Indian Chem. Soc.*, **34**, 266 (1957). [c] R. L. Frank, P. G. Arvan, J. W. Richter, and C. R. Vanneman, *J. Amer. Chem. Soc.*, **66**, 4 (1944). [d] This work; see Experimental Section. [e] Reference 12.

the unique formation of **3** can possibly be explained by assuming exclusive reduction of cation **6**, even if it arises, to **5** by iron ion as follows: $Fe^{2+} - e \rightarrow Fe^{3+}$. On the other hand, it is possible that the significant difference between carbon and platinum electrodes may be due to the presence of paramagnetic centers on the carbon anode which entirely adsorb any radicals formed.[2a] The results shown in Tables I (run 3) and II are insufficient to give definitive explanations for the formation of **2**; however, it may be tentatively concluded that coupling of **5** with hydrogen atoms occurs on the carbon electrode before desorption.

During the electrolysis of **1** in triethylamine–pyridine–water using platinum electrodes, evolution of carbon dioxide was observed on the anode surface when the applied voltage reached 60 V (cell voltage 1.58–1.59 *vs.* sce, Table IV). In this reaction, exclusive formation of α,β-unsaturated lactone **3** from **6** should be assisted by abstraction of hydrogen atom at the α position by amines.

Electrolytic decarboxylation of acid **7** to butenolide **8** was carried out successfully; subsequent hydrogenation gave in good yield the corresponding saturated lactone[7]

**Table III**
**Electrosynthesis of Δ²-Butenolides (3) from Paraconic Acids (1)**

| Paraconic acids (1) $R_1$ | $R_2$ | Registry no. | Butenolides (3) Bp (mp), °C (mm) | Yield,[a] % | Registry no. | Formula | Elemental analysis, % Calcd C | Calcd H | Found C | Found H |
|---|---|---|---|---|---|---|---|---|---|---|
| Me | Me[b] | 79-91-4 | 89–91 (1) | 68[h] | 20019-64-1 | $C_6H_8O_2$ | | | | |
| Me | $n$-$C_3H_7$[c] | 51820-72-5 | 105–107 (1) | 73 | 51820-73-6 | $C_8H_{12}O_2$ | 68.55 | 8.63 | 68.53 | 8.46 |
| Me | $n$-$C_6H_{13}$[c] | 38840-98-1 | 130–131 (1) | 85 | 51820-74-7 | $C_{11}H_{18}O_2$ | 72.49 | 9.95 | 72.64 | 9.80 |
| H | $n$-$C_6H_{13}$[d] | 20597-52-8 | 105–104 (2) | 70[i] | 2518-53-8 | $C_{10}H_{16}O_2$ | | | | |
| $-(CH_2)_4-$[e] | | 18363-10-5 | 112–113 (1) | 77 | 5732-90-1 | $C_8H_{10}O_2$ | 69.55 | 7.30 | 69.32 | 7.45 |
| $-(CH_2)_5-$[f] | | 2819-56-9 | 117–119 (2) | 78[j] | 4435-19-2 | $C_9H_{12}O_2$ | | | | |
| $-(CH_2)_{11}-$[g] | | 15210-24-9 | (85.0–86.3) | 90 | 51820-75-8 | $C_{15}H_{24}O_2$ | 76.23 | 10.24 | 76.14 | 10.31 |

[a] Yields are calculated on isolated product. [b] R. Fittig and B. Frost, *Justus Liebigs Ann. Chem.*, **226**, 370 (1884). [c] See ref 11. [d] R. Fittig and A. Schneegans, *Justus Liebigs Ann. Chem.*, **227**, 79 (1885). [e] See ref 10. The paraconic acid used in this experiment was obtained in the manner described in the Experimental Section. [f] S. F. Birch, W. Henry, and G. Armand. *J. Chem. Soc.*, **119**, 1315 (1921). [g] H. Nozaki, T. Mori, R. Noyori, and M. Kawanishi, *Can. J. Chem.*, **45**, 1804 (1967). [h] R. Fittig and C. Geisler, *Justus Liebigs Ann. Chem.*, **208**, 37 (1881). [i] K. B. Sharpless, R. F. Lauer, and A. Y. Teranishi, *J. Amer. Chem. Soc.*, **95**, 6137 (1973). [j] See ref 13.

solution and/or replacement of electrodes from platinum plates to carbon rods were found to be the most critical factors in obtaining butyrolactones (runs 2 and 3). The results are shown in Table II.

A further remarkable change in the electrolytic reaction was observed when a mixed solvent of triethylamine–pyridine–water (run 4)[5] was employed instead of the methanol–sodium methoxide solution. The results of the latter experiment, giving butenolides (**3**), are listed in Table III.

In the course of the electrolytic oxidation of **1**, either a radical or a cation intermediate such as **5** and **6** must be produced. A notable feature in the electrolysis of **1** in a mixed solvent of triethylamine–pyridine–water(run 4) is the exclusive formation of **3** *via* the cation intermediate **6**, whereas formation of **2** from **1** (runs **2** and **3**) can be considered to arise from the radical intermediate **5** followed by abstraction of hydrogen from the medium.

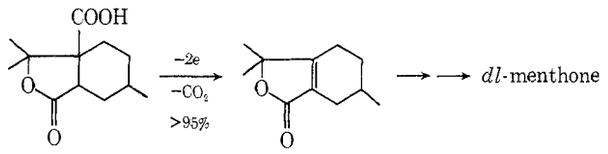
Although no evidence has been found for a catalytic effect of iron ion or iron metal in the electrolytic solution,[6]

**Table IV**
**Electrolytic Voltages in Triethylamine–Pyridine–Water Solution**

| Applied voltage, V | Cell voltage,[a] V |
|---|---|
| 30 | 1.38 |
| 40 | 1.43–1.44 |
| 50 | 1.50–1.51 |
| 60 | 1.58–1.59 |
| 70 | 1.63–1.66 |
| 80 | 1.69–1.71 |
| 90 | 1.76–1.78 |

[a] Platinum electrodes (*vs.* sce).

which can serve as a precursor for the preparation of *dl*-menthone.




7 8

## Experimental Section[8]

**Electrolysis Apparatus.** Type I consisted of two smooth platinum electrodes (3 $cm^2$) which were placed parallel to each other 2 mm apart. The electrolysis cell was a water-jacketed beaker, 3.2 cm in diameter and 10 cm high, fitted with a gas lead pipe, a thermometer, and a magnetic stirrer.[9] Current was controlled by manually adjusting the applied voltage as required. The direction of current was changed every 30 sec by means of a commutator. Type II consisted of two carbon rods cut perpendicularly into two parts (10 mm in diameter and 10 cm long), being immersed into a electrolytic solution in a depth of 5–6 cm and placed parallel to each other 3 mm apart.

**γ,γ-Tetramethyleneparaconic Acid [1, $R_1$, $R_2$ = $-(CH_2)_4-$].** A solution of cyclopentanone (3.54 ml, 0.04 mol) and zinc powder (8.16 g, 0.125 mol) in dry tetrahydrofuran (10 ml) was refluxed for 30 min. To this solution iodine (5 mg) was added and the mixture was stirred for several minutes. To the mixture methyl α-bromosuccinate in benzene (10 ml) was added dropwise over a period of 20 min. When the addition was completed, the mixture was heated to maintain gentle reflux for 2 hr. After cooling, the excess zinc powder was decomposed by dropwise addition of 20 ml of 10% aqueous acetic acid. The organic layer was separated and the aqueous layer was extracted with ether. The combined organic phases were washed with water, 10% aqueous ammonium hydroxide, dilute aqueous hydrogen chloride, and water, and dried ($Na_2SO_4$). Removal of the solvent gave an oil (7.7 g), bp 130–150° (3 mm). Without further purification, the oil was subjected to hydrolysis with excess methanolic potassium hydroxide at room temperature, after work-up as an usual manner, to give 3 g of 1 [$R_1$, $R_2$ = $-(CH_2)_4-$]: mp 133.0–134.5° (lit.[10] mp 134°); ir (Nujol) 3300–3000 (COOH), 1750 $cm^{-1}$ (C=O).[11]

**Methyl γ,γ-tetramethyleneparaconate,** mp 35.5–36.0°, was obtained by esterification of 1 [$R_1$, $R_2$ = $-(CH_2)_4-$] with diazomethane: ir (neat) 1768 (lactone C=O), 1740 $cm^{-1}$ (ester C=O); nmr ($CDCl_3$) δ 1.60–2.10 (m, 8 H, $CH_2$), 2.50–2.90 (m, 2 H, $CH_2C$=O), 3.10–3.50 (m, 1 H, CHC=O), 3.70 (s, 3 H, $CH_3O$).

*Anal.* Calcd for $C_{10}H_{14}O_4$: C, 60.59; H, 7.12. Found: C, 60.50; H, 7.15.

**Preparation of γ-Substituted γ-Butyrolactones (2). Electrolytic Procedure I (Table I, Run 2).** A solution of 1 [$R_1$, $R_2$ = $-(CH_2)_5-$] (300 mg, 1.5 mmol), sodium methoxide freshly prepared from sodium metal (400 mg, 17.4 mg-atoms), and iron powder (100 mg), after being dried up in an oven at 100° for 1 hr in dry methanol (40 ml), was electrolyzed (apparatus type I) without separation of electrolytic cells at a current of 1.25 A at 20° for 12 hr. The reaction mixture was diluted with water (50 ml) and most of the methanol was removed in a rotary evaporator. The aqueous solution was extracted with ether. The extracts were washed with diluted mineral acid and water, dried ($Na_2SO_4$), and concentrated. The residue was chromatographed over silica gel with *n*-hexane–ether (4:1) to afford 190 mg (80%) of 2 [$R_1$, $R_2$ = $-(CH_2)_5-$], bp 110–112° (1 mm) [lit.[12] bp 50° (0.05 mm)], whose spectral data are identical with those of an authentic sample.

**Electrolytic Procedure II (Table I, Run 3).** The same mixed solution described in Table I (run 2), without addition of iron powder, was electrolyzed using carbon electrodes (apparatus type II) at a current of 1.4–1.5 A at 25–35° for 20 hr. The crude product was chromatographed over silica gel with *n*-hexane–ether (4:1) to give 230 mg of 2 [$R_1$, $R_2$ = $-(CH_2)_5-$] in a quantitative yield.

In the similar manner, electrolysis of 1 [$R_1$, $R_2$ = $-(CH_2)_4-$] gave 2 [$R_1$, $R_2$ = $-(CH_2)_4-$] in 98% yield: bp 104–106° (1 mm); ir (neat) 1770 (C=O), 1163 $cm^{-1}$; mass spectrum (70 eV) *m/e* (rel intensity) 140 (23, $M^+$), 111 (100), 98 (78), 85 (20), 83 (22).

*Anal.* Calcd for $C_8H_{12}O_2$: C, 68.55; H, 8.63. Found: C, 68.55; H, 8.56.

**Preparation of $\Delta^2$-Butenolide [3, $R_1$, $R_2$ = $-(CH_2)_5-$] (Table I, Run 4).** A stirred solution of 1 [$R_1$, $R_2$ = $-(CH_2)_5-$] (300 mg, 1.5 mmol), triethylamine (30 mg), and water (4 ml) in pyridine (30 ml) was electrolyzed using carbon rods as electrodes (apparatus type II) at a current of 0.25–0.10 A at 20° for 12 hr. The reaction mixture was concentrated in a rotary evaporator. The residue was taken up in benzene (40 ml). The benzene solution was washed with aqueous 10% hydrogen chloride, followed with aqueous sodium hydrogen carbonate and water, and dried ($Na_2SO_4$). Removal of the solvent gave a crude oil (190 mg), which was purified by a capillary distillation to give 180 mg (78%) of 3 [$R_1$, $R_2$ = $-(CH_2)_5-$]: bp 117–119° (2 mm) [lit.[13] bp 84° (0.1 mm)]; ir (neat) 3070 (HC=C), 1770, 1756 (C=O), 1607 $cm^{-1}$ (C=C); nmr ($CDCl_3$) δ 1.68 (broad, 10 H, $CH_2$), 6.00 (d, 1 H, $J$ = 6 Hz, HC=C), 7.47 (d, 1 H, $J$ = 6 Hz, HC=C); mass spectrum (70 eV) *m/e* 152 ($M^+$).

***dl*-3-Carboxy-8-hydroxy-$\Delta^3$-menthene γ-Lactone (8).** A stirred solution of *dl*-3,4-dicarboxy-8-hydroxymenthane γ-lactone (7, 500 mg, 2.21 mmol), triethylamine (30 mg, 0.3 mmol), and water (4 ml) in pyridine (30 ml) was electrolyzed (apparatus type II) at a current of 0.20 A (terminal voltage 50 V) at 25–30° for 5 hr. The reaction mixture was concentrated under diminished pressure. After work-up in the usual manner, the residue was distilled to give 380 mg (95%) of 8: bp 128–129° (3 mm); mp 52–53°; ir (neat) 1755 (lactone C=O), 1675 $cm^{-1}$ (C=C); nmr ($CDCl_3$) δ 1.09 (d, 3 H, $J$ = 6 Hz, $CH_3$), 1.42 (s, 6 H, $CH_3$); mass spectrum (70 eV) *m/e* 180 ($M^+$).

*Anal.* Calcd for $C_{11}H_{16}O_2$: C, 73.30; H, 8.95. Found: C, 73.32; H, 8.82.

**Registry No.**—7, 51820-76-9; 8, 51820-77-0; methyl γ,γ-tetramethyleneparaconate, 18363-04-7; cyclopentanone, 120-92-3; methyl α-bromosuccinate, 760-90-7.

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