Rodlike Molecules by Kolbe Electrolysis

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A new short and simple pathway to rigid, rod-shaped hydrocarbon skeletons, in particular of the oligo-bicyclo[2.2.2]octane type, is described. The key step consists of an electrochemical C–C bond coupling reaction between bridgehead positions of bi- and tricyclic carboxylic acids. Functional groups can be retained, they influence the yield of the C–C bond connection. In this way, otherwise difficult or laborious syntheses are shortened, and rigid, non-collapsable nano size spacer units are easily available. The optimized electrochemical procedures are described in detail.

For various applications, such as the synthesis of non-collapsable nano size molecules,[1] liquid crystals[2] and the kinetic determination of intramolecular electron and energy transfer reactions,[3] rigid, rodlike molecules are advantageous.[4] In most cases, the construction of such molecules demands a large number of steps, which can not be performed in a repetitive manner.[5] Additionally, the methodology is not flexible enough and less suitable for constructing longer rods (an exception is the method of Ayres et al.[4]).

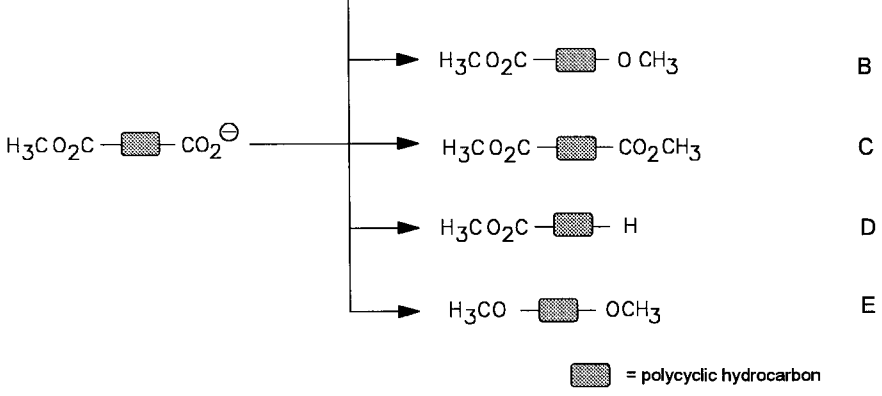
We describe here a new strategy for the preparation of rodlike substances via electrochemical coupling of two carboxylic acid molecules, based on the Kolbe or in special cases the Brown–Walker C–C coupling.[6] Up to now, this coupling was mainly used to connect carboxylic acids with one or two alkyl substituents in the α-position.[7] In this context, we were successful in coupling bi- and tricyclic carboxylic acids, which are highly branched in the α-position (bridgehead carbons).

Yield and selectivity of the Kolbe synthesis[8] are strongly dependent on the selection of the structure of the acid and on the reaction conditions. The parameters influencing the reaction are current density, pH, temperature, additives, solvent, supporting electrolyte and electrode material. A high current density and a resulting high concentration of alkyl radicals at the electrode surface favour the generation of dimeric products. Higher temperatures suppress the coupling and the competing disproportionation reaction is favoured.[9] A carboxylate layer that forms at the electrode surface appears to be necessary for high dimer yields. Additional anions besides the carboxylate and most electrode materials except platinum prevent the generation of this layer and therefore they should be strictly excluded. On the other hand, the layer can be supported by the choice of the solvent. Common solvents are methanol and methanol/water, but sometimes dioxane, dimethylacetamide, acetonitrile/water or dimethylsulfoxide is used. It is advantageous to use weakly acidic solutions,[8] with only partial neutralization of the acid.

In our case, two Pt-foil electrodes were fixed in a three necked flask equipped with a reflux condenser. The best results were achieved by using a current density of approximate 0.7 A/cm^2 (voltage of 300–400 V is necessary; see experimental part), methanol as solvent and a saturated, 10% neutralized solution of the acid. During the reaction, the mixture should be cooled (– 20 °C) to avoid side reactions (Scheme 1).

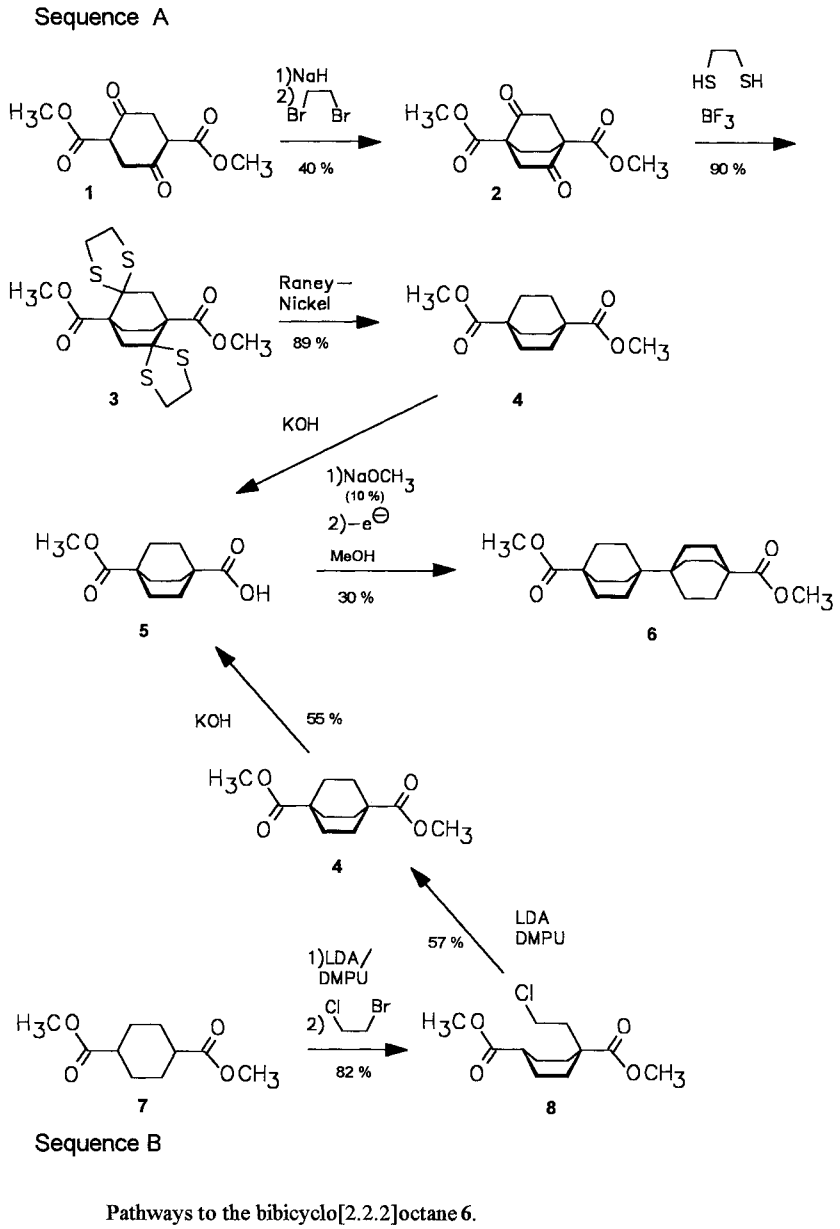
The yields of the reactions are quite acceptable, especially if they are compared with the methods applied hitherto,[10] which often need more steps.

Thus, dimethyl 1,1′-bibicyclo[2.2.2]octane-4,4′-dicarb-



Products of the Brown–Walker reaction in methanol: A = Brown–Walker product, B = Hofer–Moest product, C = acidic esterification, D = decarboxylation, E = hydrolysis of the ester followed by double Hofer–Moest reaction

Scheme 1



Pathways to the bibicyclo[2.2.2]octane 6.

Scheme 2

Table 1. Product Distribution in the Kolbe Electrolysis of the Bicyclooctanecarboxylic Acid **5**[a]

Electrodes	*T* of Cooling Bath (°C)	Concentration (mol/l)	Yield (%) A	B	C	D	E
Pt-wire electrodes	0	0.786	10.6	43.7	22.4	9.7	8.1
Pt-wire anode – carbon cathode	0	0.786	5.2	35.1	25.6	9.1	12.7
Pt-wire cathode – Pt-foil anode	0	0.786	15.8	47.9	17.5	6.2	8.8
Pt-foil electrodes	0	0.354	20	0	8.8	33.7	37.1
Pt-foil electrodes	0	0.707	28.4	0	18.3	13.5	36.4
Pt-foil electrodes	−20	2.95	29.8	19.9	11.2	7.4	27.4

[a] See Scheme 1. Polycyclic hydrocarbon = bicyclo[2.2.2]octane; yields were determined by GC.

oxylate **(6)** (product A in Scheme 1) could be synthesized in four steps (Scheme 2), and 30% of the starting material was coupled to the corresponding dimer (Table 1).

The solubility of rigid molecules decreases with increasing length of the compound. Thus, the dimer **6** precipitated and separation was achieved by filtration. To obtain **6** using the common methods (dimerization of iodine derivatives by use of nickel or sodium), at least 8 steps have to be carried out to isolate the coupling precursor (8.9% yield over all steps of Sequence B, Scheme 2, compared with 4.4% yield over all steps to isolate the coupling precursor).[10]

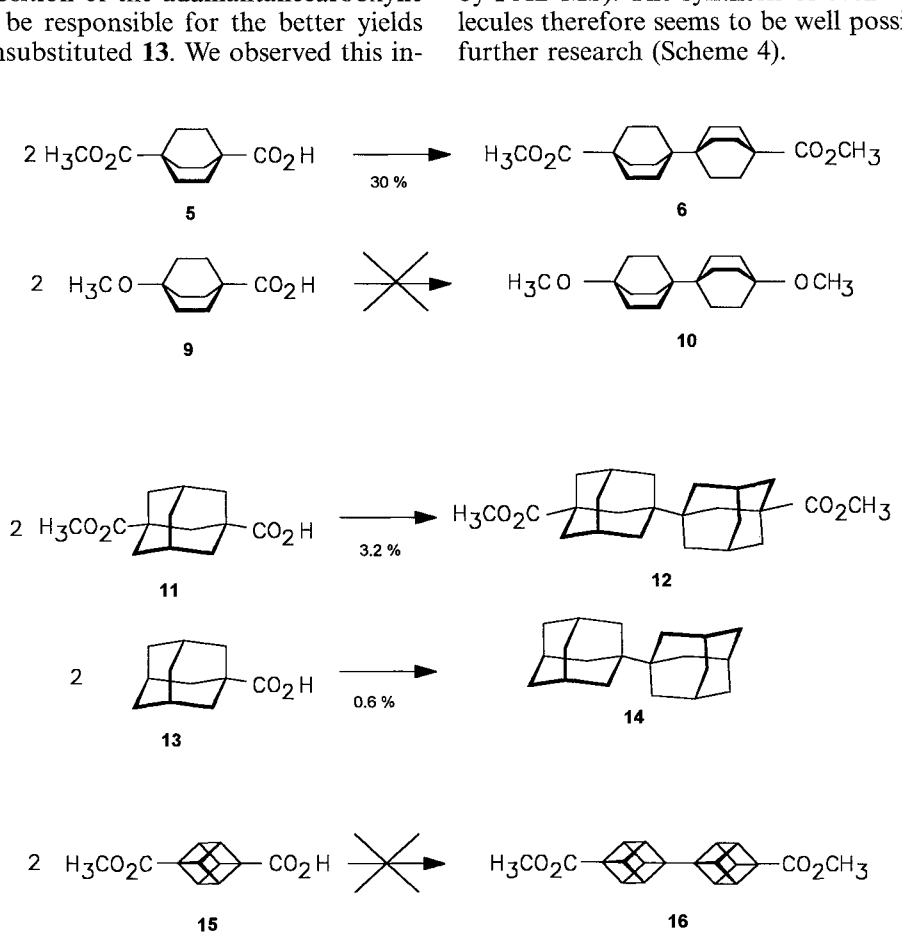
Other bi- and tricyclic carboxylic acids reacted in the same way (Scheme 3).[11]

According to Van Zorge et al., the Kolbe electrolysis of 1-adamantanecarboxylic acid **(13)** has been suspected to be unsuccessful, because formation of the Hofer–Moest product[12] (cf. Scheme 1, B) is favoured.[4] Besides the main product (the Hofer–Moest product), in contrast to these earlier findings, we were able to dimerize not only adamantane-1,3-dicarboxylic acid monomethyl ester **(11)** under the conditions mentioned above to form the bisadamantane derivative **12**, but also the acid **13**, albeit in small amounts.[13] Besides the small amount of the biadamantane **14**, an isomer with the same molecular mass was generated.[14] In compound **11**, the carboxylic ester function in the 3-position of the adamantanecarboxylic acid **(11)** seems to be responsible for the better yields compared to the unsubstituted **13**. We observed this influence in another case also: in the electrolysis of 4-methoxy-1-bicyclo[2.2.2]octane **(9)**, no coupling product could be detected, although satisfactory yields of **6** were obtained starting from **5** (Scheme 3). This could be due to through-space or through-bond interactions.[15] Electron-withdrawing groups (and electron-donating groups) in the 4-position of the bicyclo[2.2.2]octane skeleton influence the acid strength of a carboxylic group in the 1-position;[16] the same phenomenon was observed in the adamantane series.[17] These groups could also disturb the generation and stability of the radicals, which are the precursors of the dimer.

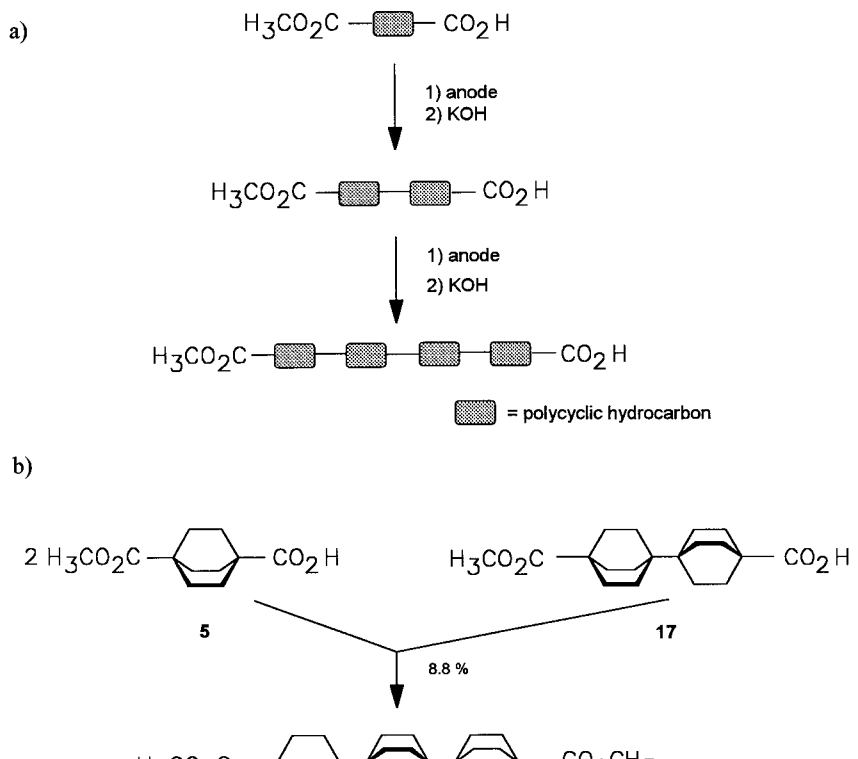
The framework of the carboxylic acid also influences the yield of the dimerization: with dimethyl cubane-1,4-dicarboxylate **(15)** we could not find any dimer (the carboxylic acid was mainly transformed to the corresponding methyl ester).

Besides the simplicity of these reactions our new strategy has the advantage of allowing repetition of the crucial steps (cleavage of the diester and coupling reaction) to end up with longer molecules in another Kolbe or cross-Kolbe reaction.

We also isolated 8.8% of the terbicyclo[2.2.2]octane **18** by a cross-Kolbe electrolysis of bicyclo- **(5)** and bibicyclo[2.2.2]octane **(17)**, which was formed besides the bicyclooctane **6** and a trace of dimethyl 1,1′:4′,1″:4″,1‴-quaterbicyclo[2.2.2]octane-4,4‴-dicarboxylate (detected by FAB-MS). The synthesis of even longer rodlike molecules therefore seems to be well possible and is part of further research (Scheme 4).



Scheme 3 *Kolbe*-reactions of polycyclic hydrocarbon skeletons.



Pathways to longer rods: a) Repetitive Kolbe reaction b) The terbicyclo[2.2.2]octane **18** is formed by cross-Kolbe reaction of **5** and **17**. Side products are bicyclo[2.2.2]octane and quaterbicyclo[2.2.2]-octane derivatives.

Scheme 4

Solvents were purified by standard methods and dried if necessary. Reagents used were commercial quality. Ni/Al-alloy (B013) was obtained by Degussa, Hanau. TLC was carried out on silica gel 60 F 254 (E. Merck, Darmstadt, Germany), detection with UV-light ($\lambda = 254$ nm) and Rhodamin 6G (solution in EtOH). Melting points were determined on a microscope heating unit of Reichert, Vienna, and are not corrected. IR spectra were recorded with a infrared spectrometer of Perkin-Elmer, 1600, FTIR, Connecticut, USA. The NMR spectra were measured on WP-60 (^{1}H: 60 MHz) of Varian Associates, Paolo Alto, USA and on WM-250 (^{1}H: 250 MHz, ^{13}C: 63 MHz) and AM-400 (^{1}H: 400 MHz; ^{13}C: 100 MHz) and DRX 500 (^{1}H: 500 MHz, ^{13}C: 125 MHz) of Bruker Physik AG, Karlsruhe.[18] EI-MS were performed with MS-50 of A.E.I., Manchester, GC/MS and GC with a unit of Hewlett Packard [GC: HP 5890, Series 2; MS: HP 5989A; HP1] [capillary column (12 m), "cross-linked methyl-silicon"].[19] The power supply (galvanostat) was manufactured by the electronic department of the Chemical Institute of the University of Bonn, max. wattage 450 V × 1.5 A.

Sequence **B** (Scheme 2) was performed according to the method of Della et al.[20] The other pathway (Sequence **A**, Scheme 2) was examined following a procedure of Roberts et al.[21] 4-Methoxybicyclo[2.2.2]octane-1-carboxylic acid **(9)** was synthesized according to Adcock et al..[22]

Dimethyl 1,4-Dioxobicyclo[2.2.2]octane-2,5-dicarboxylate (2):
NaH (118 g, 3.93 mol, 80% suspension in oil) and dimethyl 1,4-dioxocyclohexane-2,5-dicarboxylate **(1)** (224.75 g, 0.99 mol) were added in frequent portions to 1 L 1,2-dimethoxyethane, the mixture was stirred with a KPG-stirrer. Afterwards, NaH was added to the suspension until the evolution of hydrogen ceased. Then 75% of the solvent was distilled off. During the distillation the colour of the suspension changed from grey-yellow to pink. After cooling the mixture, 760 mL of 1,2-dibromoethane was added dropwise. The suspension was then heated until the pink colour disappeared. The hot solution was filtered and the solid was washed twice using CH_2Cl_2. The solvent and the excess of dibromoethane were removed from the filtrate by vacuum distillation. The yellow oil was poured into a small amount of MeOH. The yellow crude product was recrystallized and separated. Recrystallization from MeOH gave 100.8 g (40%) of a colourless solid with a mp of 150–152°C.

MS (EI, 70 eV): m/z (%) = 254 (M^+, 10), 222 ($M-CH_4O^+$, 40), 195 ($M-CH_4O-C_2H_5^+$, 23).

^{1}H NMR (60 MHz, $CDCl_3$): $\delta = 2.2–2.5$ (m, 4H, CH_2), 2.8–3.2 (m, 4H, CH_2CO), 3.7 (s, 6H, OCH_3).

Dimethyl 2,5-Bi[1,3]dithiolanylbicyclo[2.2.2]octane-1,4-dicarboxylate (3):
25.6 g (0.1 mol) Dimethyl 2,5-dioxobicyclo[2.2.2]octane-1,4-dicarboxylate **(2)** was dissolved in 110 mL glacial AcOH 25.3 mL (0.2 mol) $BF_3 \cdot OEt_2$ complex followed by 23.2 mL (0.27 mol) 1,2-ethanedithiol were added and the solution was stirred for 24 h. A yellowish solid precipitated and was extracted with $CHCl_3$. The organic layer was separated, washed with water (3 × 20 mL), aq $NaHCO_3$ (5%) and water. The layer was dried (Na_2SO_4), the solvent evaporated and the oily product was poured into MeOH 36.1 g (88.9%) of a colourless solid (mp 84–86°C) crystallized.

MS (EI): m/z (%) = 406 (M^+, 100), 374 ($M-OCH_2^+$, 60), 301 ($M-OCH_2-C_3H_5O_2^+$, 55), 228 (30), 189 ($M-2\times S_2C_2H_4^+$, 80).

^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 1.1$ (s, CH_2), 1.8–1.95 (m), 2.3–3.3 (m), 3.35 (s), 3.7 (s, OCH_3).

^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 26.38$ (s, 2 C, CH_2), 39.39 (s, 2 C, CH_2S), 40.56 (s, 2 C, CH_2S), 50.46 (s, 2 C, C_q), 51.16 (s, 2 C, CH_2), 52.09 (s, 2C, OCH_3), 69.97 (s, 2C, C_q), 172.98 (s, 2C, CO_2CH_3).

Dimethyl Bicyclo[2.2.2]octane-1,4-dicarboxylate (4):
20.00 g (0.049 mol) of the thioketal **3** was added to a suspension of 300 g Raney nickel catalyst (activity W2[23]) in 500 mL EtOH. This suspension was heated for 4 d. Then the nickel was filtered off hot and washed twice with hot EtOH. The solvent was then evaporated and the colourless solid recrystallized from MeOH to give 9.88 g (89.2%) of **4**, mp = 95–98 °C.

MS (EI): *m/z* (%) = 226 (M^+, 10), 194 ($M-CH_4O^+$, 12), 135 ($M-CH_4O-C_2H_3O_2^+$, 45), 107 ($M-2\times C_2H_3O_2^+$, 100).

^{1}H NMR (60 MHz, $CDCl_3$): δ = 1.8 (s, 12H, CH_2), 3.6 (s, 6H, OCH_3).

Partial Hydrolysis of the Diesters; General Procedure:
The diester and the equivalent amount of KOH were dissolved in MeOH (95%). The solution was heated for the period given below. Afterwards the solvent was evaporated. The residue was poured into water and this solution was extracted with diethyl ether to remove the starting material. The aqueous solution was acidified with aq HCl (to pH 4) and extracted three times with CH_2Cl_2. The organic layer was filtered, dried (Na_2SO_4) and then the solvent was evaporated.

Monomethyl Bicyclo[2.2.2]octane-1,4-dicarboxylate **(5)**:
9.80 g (49 mmol) Dimethyl bicyclo[2.2.2]octane-1,4-dicarboxylate **(4)**; 2.5 g (44 mmol) KOH; 250 mL MeOH (95%), 1 d reaction time. Yield: 4.64 g (44.9%) of **5**, a colourless solid; mp 176 °C.

HRMS: *m/z* calc. for $C_{11}H_{16}O_4$ 212.1049; found, 212.1047.

MS (70 eV): *m/z* (%) = 212 (M^+, 95), 183 ($M-29^+$, 8), 180 ($M-CH_4O^+$, 70), 153 ($M-C_2H_3O_2^+$, 40), 135 ($M-CO_2CH_3-H_2O^+$, 47), 107 ($M-C_3H_4O_4^+$, 100).

^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.5–2.0 (s, 12H, CH_2), 3.6 (s, 3H, CH_3), 10.5 (s, 1H, CO_2H).

^{13}C NMR (63 MHz, $CDCl_3$): δ = 27.64 (s, 3C, CH_2), 27.72 (s, 3C, CH_2), 38.6 (s, 1C, C_q), 38.7 (s, 1C, C_q), 51.88 (s, 1C, OCH_3), 177.88 (s, 1C, CO_2CH_3), 184 (s, 1C, CO_2H).

Monomethyl 1,1'-Bibicyclo[2.2.2]octane-4,4'-dicarboxylate **(17)**:
496 mg (1.48 mmol) Dimethyl 1,1'-bibicyclo[2.2.2]octane-4,4'-dicarboxylate **(6)**, 97.3 mg (1.48 mmol) KOH (85%), 250 mL MeOH (95%), 6 d reaction time. Yield: 196.4 mg (42%) of a colourless solid, mp 320 °C.

HRMS: *m/z* calc. for $C_{19}H_{28}O_4$, 320.1987; found, 320.1987.

^{1}H NMR (250 MHz, $CDCl_3/CD_3OD$): δ = 1.25 (m, 12H, CH_2), 1.6 (m, 12H, CH_2), 3.5 (s, 3H, OCH_3).

^{13}C NMR (63 MHz, $CDCl_3$): δ = 24.49 (s, 6C, CH_2), 28.2 (s, 6C, CH_2), 34.5 (s, 2C, C_q), 38.2 (s, 2C, C_q), 51.6 (s, 1C, OCH_3), 179.3 (s, 1C, CO_2CH_3), 181.2 (s, 1C, CO_2H).

Monomethyl 1,3-Adamantanedicarboxylate **(11)**:[17]
16.3 g (64.6 mmol) Dimethyl 1,3-adamantanedicarboxylate, which was prepared according to Landa et al.,[24] 3.62 g (64.6 mmol) KOH, 200 mL MeOH (95%). 1 d reaction time. Yield 10.73 g (69.7%) of **11** as a colourless solid, mp 110 °C.

MS (70 eV): *m/z* % = 238 (M^+, 25), 193 ($M-CH_2O^+$, 64), 179 ($M-CO_2CH_3^+$, 100), 161 ($179-H_2O^+$, 34), 133 ($M-CH_2O-CO_2CH_3-H^+$, 67), 105 ($133-C_2H_4^+$, 18), 91 ($133-C_3H_6^+$, 35), 79 ($133-2\times C_2H_4^+$, 25).

^{1}H NMR (60 MHz, CD_3OD): δ = 1.6–2.1 (14H, CH_2, CH), 3.6 (s, 3H, OCH_3), 9.6–10.0 (s br, 1H, CO_2H).

Monomethyl Cubane-1,4-dicarboxylate **(15)**:[25]
750 mg (3.4 mmol) Dimethyl cubane-1,4-dicarboxylate (commercially available), 224 mg (3.4 mmol) KOH (85%), 250 mL MeOH (95%), 4 d reaction time. Yield, 452.31 mg (64.6%) of **15** as a colourless solid, mp 175 °C.

^{1}H NMR (250 MHz, CD_3OD): δ = 3.6 (s, 3H, OCH_3), 4.04 (s, 6H, CH).

^{13}C NMR (63 MHz, $CDCl_3$): δ = 47.1 (s, 3C, CH), 51.7 (s, 1C, OCH_3), 55.6 (s, 1C, C_q), 55.84 (s, 1C, C_q), 172.05 (s, 1C, $CO_2CH_3CH_3$), 177.68 (s, 1C, CO_2H).

Electrolysis; General Procedure:
In a three-necked flask (10 mL) equipped with a reflux condenser, the acid was dissolved in MeOH and 10% of the acid was neutralized with NaOMe. This solution was electrolized using Pt-foil electrodes (each 0.24 cm^2) fixed in two of the necks of the flask (distance between anode and cathode: 1 cm, voltage of 300–400 V and a fixed current density of 0.7 A/cm^2; the time is given below). Electrolysis with considerably lower voltage should be possible by using special cells with a smaller distance between the electrodes, such as the flow-through cell with a 0.5 mm electrode gap mentioned in the literature.[26]

After the start and during the electrolysis, the solution was cooled with solid CO_2/EtOH (bath temperature −20 °C). Purification is described below for each compound.

Dimethyl 1,1'-Bibicyclo[2.2.2]octane-4,4'-dicarboxylate **(6)**:
2.5 g (0.0118 mol) Monomethyl bicyclo[2.2.2]octane-1,4-dicarboxylate **(5)** in 4 mL MeOH, duration 5 h. The product, a colourless solid, was filtered off and washed twice with MeOH. Yield: 0.589 g (29.8%); mp 229 °C.

MS (EI) *m/z* (%) = 334 (M^+, 10), 305 ($M-29^+$, 3), 274 ($M-CO_2CH_3-H^+$, 100), 245 ($M-CO_2CH_3-CH_2O^+$, 4), 215 (M-2 $CO_2CH_3-H^+$, 22), 167 ($M-C_{10}H_{15}O_2^+$, 25), 139 (18), 107 (50), 79 (35), 59 ($CO_2CH_3^+$, 15).

Anal. ($C_{20}H_{30}O_4$): Calc. C, 71.82; H, 9.04. Found C, 71.85; H, 9.28.

FT-IR (KBr): ν = 2950.9 s, 2865.9 m, 1718.6 s, 1256.2 s, 1226.4 s, 1069.6 s cm^{-1}.

^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.35 (ddd, 12H, CH_2), 1.65 (ddd, 12H, CH_2), 3.58 (s, 6H, OCH_3).

^{13}C NMR (63 MHz, $CDCl_3$): δ = 24.8 (s, 6C, CH_2), DEPT −, 28.2 (s, 6C, CH_2) DEPT −, 34.8 (s, 2C, Cq), 38.4 (s, 2C, Cq), 51.7 (s, 2C, OCH_3), DEPT +, 178.9 (s, 2C, CO_2CH_3).

Dimethyl 1,1':4'1''-Terbicyclo[2.2.2]octane-4,4''-dicarboxylate **(18)**:
Electrolysis was performed as before. 400 mg (1.89 mmol) Monomethyl bicyclo[2.2.2]octane-1,4-dicarboxylate **(5)** and 90 mg (0.28 mmol) monomethyl 1,1'-bibicyclo[2.2.2]octane-4,4'-dicarboxylate **(17)** were dissolved in MeOH. After electrolysis (40 min), 45 mg of a colourless solid was filtered off and washed with MeOH. This solid was a mixture of **18** and **6**. In this mixture, a trace of dimethyl 1,1':4',1'':4'',1'''-quaterbicyclo[2.2.2]octane-4,4'''-dicarboxylate [FAB-MS: *m/z* = 551 ($M+H^+$)] was detected, which was not isolated. Purification was performed by chromatography (silica gel; CH_2Cl_2, R_f = 0.38). Yield of **18** 10.84 mg (8.8%), mp > 280 °C.

HRMS: *m/z* calc. for $C_{28}H_{42}O_4$, 442.3083; found, 442.3083.

FAB-MS: *m/z* (%) = 443 ($M+H^+$, 100).

MS (EI): *m/z* (%) = 442.2 (M^+, 5), 382 ($M-2\times CH_2O^+$, 20), 274 (100).

FT-IR (KBr): ν = 2942.1 s, 2850 w, 1734 s, 1600 m br cm^{-1}.

^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.25 (s, 12H, CH_2), 1.3–1.38 (m, 12H, CH_2), 1.65–1.75 (m, 12H, CH_2), 3.6 (s, 6H, OCH_3).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 24.8 (s, 6C, CH_2), 25.7 (s, 6C, CH_2), 28.9 (s, 6C, CH_2), 34.2 (s, 2C, C_q), 35.0 (s, 2C, C_q), 38.8 (s, 2C, C_q), 52.1 (s, 2C, OCH_3), 184.0 (s, 2C, CO_2CH_3).

Dimethyl 1,1'-Biadamantane-3,3'-dicarboxylate **(12)**:
3 g Monomethyl 1,3-adamantanedicarboxylate **(11)**, 20 mg NaOMe, 4 mL MeOH, duration 6 h. The solvent was evaporated followed by chromatography (silica gel; CH_2Cl_2, R_f = 0.27) of the residue. The product was obtained as a colourless solid. Yield 78.3 mg (3.2%) of **12**, mp 141 °C.

MS (EI): *m/z* (%) = 386 (M^+, 20), 327 (5), 193 (M^{2+}, 100), 161 (30), 133 (75).

Anal. ($C_{24}H_{34}O_4$): Calc. C, 74.58; H, 8.87. Found C, 74.16; H, 9.02.

^{1}H NMR (250 MHz, $CDCl_3$): δ = 1.49–1.6 (m, 12H, CH_2), 1.65 (s br, 4H, CH_2), 1.75 (m, 8H, CH_2), 2.08 (m, 4H, CH), 3.61 (s, 6H, OCH_3).

^{13}C NMR (63 MHz, $CDCl_3$): δ = 28.57 (s, 8C, CH_2), 34.36 (s, CH_2), 36.3 (s, CH_2), 36.94 (s), 37.0 (s), 38.65 (s, CH_2), 41.72 (s, CH_2), 51.68 (s, 2C, OCH_3), 178.4 (s, 2C, CO_2CH_3).

1,1'-Biadamantane **(14)**:

2.00 g (11 mmol) 1-Adamantanecarboxylic acid **(13)**, 10 mg NaOMe, 3 mL MeOH, duration 4 h. The solvent was evaporated followed by chromatography (silica gel; CH_2Cl_2, $R_f = 0.3$) of the residue. The product was obtained as a colourless solid. Yield 4.8 mg (0.64%) of **14**, mp 286°C.

MS (EI) *m/z* (%) = 270 (M^+, 13), 135 (M^{2+}, 100), 119 (3), 107 (10), 93 (30), 79 (35), 67 (18).

^{1}H NMR (250 MHz, $CDCl_3$): $\delta = 1.44-1.48$ (d, 12H, CH_2), 1.5–1.58 (m, 12H, CH_2), 1.8–1.86 (m, 6H, CH).

^{13}C NMR (63 MHz, $CDCl_3$): $\delta = 28.91$ (s, 6C, CH_2), 35.09 (s, 6C, CH_2), 36.21 (s, 2C, C_q), 37.47 (s, 6C, CH_2).

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